Supporting Information

Direct Oxidative Amidation between Methylarenes and Amines in Water**

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General Considerations：

All reactions were carried out in ovendried glassware. All methylarenes and amine or amine hydrochloride salts were obtained from commercial sources and used as received. All the reactions were monitored by thin-layer chromatography (TLC); products purification was done using silica gel column chromatography.

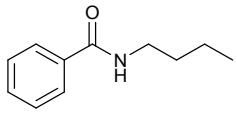
$^{1}H/^{13}C$ NMR spectra were recorded on Bruker avance 400 MHz and Bruker AMX 400 MHz spectrometer at 400/100 MHz, respectively, in $CDCl_3$ unless otherwise stated, using either TMS or the undeuterated solvent residual signal as the reference. Chemical shifts are given in ppm and are measured relative to $CDCl_3$ or DMSO-d_6 as an internal standard. Mass spectra were obtained by the electrospray ionization time-of-flight (ESI-TOF) mass spectrometry. GC yields were obtained using biphenyl as an internal standard. Flash column chromatography purification of compounds was carried out by gradient elution using ethyl acetate (EA) in light petroleum ether (PE).

General experimental procedure for secondary amides:

To a mixture of tretrabutylammonium iodide (26.6 mg, 0.072 mmol, 30 mol %), $FeCl_3 \cdot 6H_2O$ (0.0973 mg, 0.036 mmol, 15 mol %), 4 Å molecular sieves (100 mg) in water (1.5 mL) were added methylarenes **1** (4.8 mmol, 20 equiv), amine **2** (0.24 mmol), and TBHP (70 wt % in water, o.21 mL, 1.44 mmol, 6 equiv). The reaction mixture was stirred at 60 ℃ inside sealed tube for 24 h. After the reaction was completion (TLC), the contents were cooled to room temperature and then extracted with ethyl acetate (3 x 10 mL). The combined organic phase was dried over anhydrous Na_2SO_4, filered and evaporated under reduced pressure to afford the crude product which was further purified by flash chromatography on silica gel gradient with elution of ethyl acetate in petroleum ether to obtain the amide product **3.**

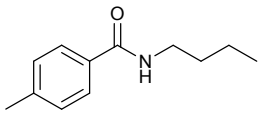
Characterization of products:

N-Butylbenzamide (3a)[1]



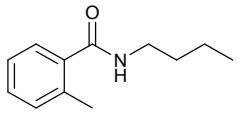
Yellow liquid; yield: 73%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.75 (d, *J* = 7.7 Hz, 2H), 7.48–7.37 (m, 3H), 6.36 (bs, 1H), 3.45–3.40 (m, 2H), 1.64–1.53 (m, 2H), 1.43–1.36 (m, 2H), 0.93 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.5, 134.9, 131.2, 128.4, 126.8, 39.8, 31.7, 20.1, 13.7.

N-Butyl-4-methylbenzamide (3b)[2]



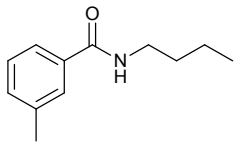
Yellow solid; yield: 78%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.65 (d, *J* = 8.2 Hz, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 6.30 (bs, 1H), 3.44–3.39 (m, 2H), 2.37 (s, 3H), 1.61–1.54 (m, 2H), 1.43–1.34 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.4, 141.5, 132.0, 129.1, 126.8, 39.7, 31.7, 21.3, 20.1, 13.7.

N-Butyl-2-methylbenzamide (3c)[2]



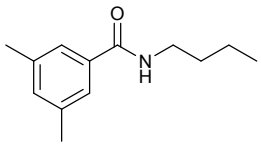
Yellow solid; yield: 50%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.33–7.27 (m, 2H), 7.21–7.16 (m, 2H), 5.82 (bs, 1H), 3.44–3.40 (m, 2H), 2.43 (s, 3H), 1.62–1.54 (m, 2H), 1.46–1.36 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 170.1, 136.8, 135.8, 130.9, 129.6, 126.6, 125.6, 39.5, 31.7, 20.1, 19.7, 13.7.

N-Butyl-3-methylbenzamide (3d)[3]



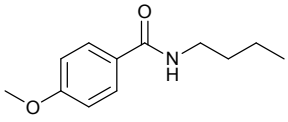
Yellow liquid; yield: 65%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.59–7.54 (m, 2H), 7.28–7.27 (m, 2H), 6.38 (bs, 1H), 3.45–3.40 (m, 2H), 2.37 (s, 3H), 1.64–1.52 (m, 2H), 1.44–1.35 (m, 2H), 0.94 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.7, 138.2, 134.8, 131.9, 128.3, 127.6, 123.7, 39.7, 31.7, 21.2, 20.1, 13.7.

N-Butyl-3,5-dimethylbenzamide (3e)[1]



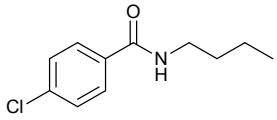
Yellow solid; yield: 65%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.35 (s, 2H), 7.08 (s, 1H), 6.29 (bs, 1H), 3.55–3.34 (m, 2H), 2.32 (s, 6H), 1.64–1.50 (m, 2H), 1.46–1.32 (m, 2H), 0.95–0.93 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.9, 138.1, 135.0, 132.7, 124.6, 39.7, 31.8, 21.1, 20.1, 13.7.

N-Butyl-4-methoxybenzamide (3f)[3]



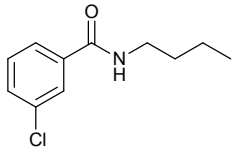
Yellow liquid; yield: 82%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.72 (d, *J* = 7.7 Hz, 2H), 6.87 (d, *J* = 7.8 Hz, 2H), 6.31 (bs, 1H), 3.81 (s, 3H), 3.53–3.29 (m, 2H), 1.58–1.52 (m, 2H), 1.40–1.34 (m, 2H), 0.92 (t, *J* = 6.7 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.0, 161.9, 128.6, 127.0, 113.5, 55.3, 39.7, 31.7, 20.1, 13.7.

N-Butyl-4-chlorobenzamide (3g)[3]



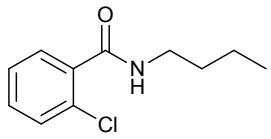
White solid; yield: 60%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.69 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 6.23 (bs, 1H), 3.45–3.40 (m, 2H), 1.62–1.54 (m, 2H), 1.44–1.35 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (101 MHz, $CDCl_3$):** δ 166.4, 137.5, 133.2, 128.8, 128.3, 39.9, 31.6, 20.1, 13.7.

N-Butyl-3-chlorobenzamide (3h)[3]



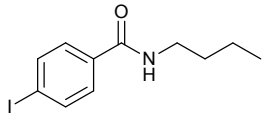
Colorless solid; yield: 69%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.73 (s, 1H), 7.62 (d, *J* = 7.7 Hz, 1H), 7.44 (d, *J* = 8.1 Hz, 1H), 7.36-7.32 (m, 1H), 6.27 (bs, 1H), 3.45–3.40 (m, 2H), 1.62–1.55 (m, 2H), 1.44–1.35 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 166.2, 136.6, 134.6, 131.3, 129.8, 127.2, 124.9, 39.9, 31.6, 20.1, 13.7.

N-Butyl-2-chlorobenzamide (3i)[4]



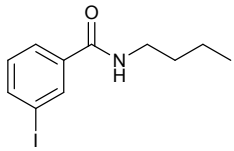
White solid; yield: 22%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.65–7.63 (m, 1H), 7.41–7.28 (m, 3H), 6.19 (s, 1H), 3.49–3.44 (m, 2H), 1.66–1.56 (m, 2H), 1.47–1.38 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 166.4, 135.3, 131.1, 130.5, 130.1 (2C), 127.1, 39.9, 31.5, 20.1, 13.7.

N-Butyl-4-iodobenzamide (3j)



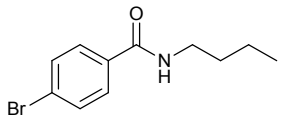
White solid; yield: 78%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.74 (d, *J* = 7.1 Hz, 2H), 7.47 (d, *J* = 7.1 Hz, 2H), 6.27 (bs, 1H), 3.43–3.39 (m, 2H), 1.62–1.52 (m, 2H), 1.45–1.33 (m, 2H), 0.94 (t, *J* = 6.8 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 166.7, 137.7, 134.3, 128.5, 98.0, 39.9, 31.7, 20.1, 13.69; IR (KBr) ν 3303, 3073, 2957, 2930, 1726, 1622,1588, 1009; HRMS (ESI-TOF) calcd for $C_{11}H_{14}INNaO$ ($M + Na^+$) 326.0018, found 326.0037.

N-Butyl-3-iodobenzamide (3k)[5]



Yellow solid; yield: 70%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.08 (s, 1H), 7.80 (d, *J* = 7.7 Hz, 1H), 7.70 (d, *J* = 7.6 Hz, 1H), 7.17-7.13 (m, 1H), 6.22 (bs, 1H), 3.45–3.40 (m, 2H), 1.62–1.52 (m, 2H), 1.44–1.37 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 166.0, 140.1, 136.8, 135.9, 130.2, 126.1, 94.2, 39.9, 31.6, 20.1, 13.7.

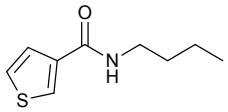
4-Bromo-N-butylbenzamide (3l)[5]



Yellow solid; yield: 74%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.62 (d, *J* = 8.5 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 6.24 (s, 1H), 3.45–3.40 (m, 2H), 1.62–1.55 (m, 2H), 1.44–1.35 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H);

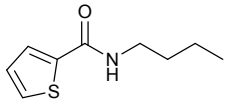
^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5, 133.8, 131.7, 128.5, 125.9, 39.9, 31.7, 20.1, 13.7.

N-Butylthiophene-3-carboxamide (3m)[6]



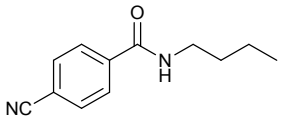
Yellow solid; yield: 50%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.86 (dd, *J* = 3.0, 1.3 Hz, 1H), 7.39 (dd, *J* = 5.1, 1.3 Hz, 1H), 7.29 (dd, *J* = 5.1, 3.0 Hz, 1H), 6.36 (bs, 1H), 3.41–3.36 (m, 2H), 1.59–1.52 (m, 2H), 1.41–1.32 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 163.2, 137.7, 127.8, 126.2, 126.1, 39.5, 31.7, 20.1, 13.7.

N-Butylthiophene-2-carboxamide (3n)[3]



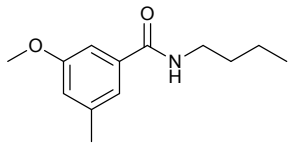
Yellow solid; yield: 55%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.49 (d, *J* = 3.4 Hz, 1H), 7.44 (d, *J* = 4.9 Hz, 1H), 7.09–6.99 (m, 1H), 6.11 (bs, 1H), 3.45–3.40 (m, 2H), 1.62–1.55 (m, 2H), 1.44–1.35 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 161.9, 139.2, 129.6, 127.8, 127.5, 39.7, 31.7, 20.1, 13.7.

N-Butyl-4-cyanobenzamide (3o)[4]



White solid; yield: 58%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 8.0 Hz, 2H), 6.40 (bs, 1H), 3.45–3.42 (m, 2H), 1.66–1.52 (m, 2H), 1.42–1.36 (m, 2H), 0.94 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 165.7, 138.7, 132.4, 127.6, 118.0, 114.8, 40.0, 31.5, 20.1, 13.7.

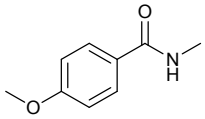
N-Butyl-3-methoxy-5-methylbenzamide (3p)



Yellow liquid; yield: 72%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.13 (s, 1H), 7.09 (s, 1H), 6.83 (s, 1H), 6.11 (s, 1H), 3.82 (s, 3H), 3.46–3.41 (m, 2H), 2.35 (s, 3H), 1.63–1.55 (m, 2H), 1.45–1.36 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.6, 159.8, 139.7, 136.2, 119.6, 118.1, 109.4,

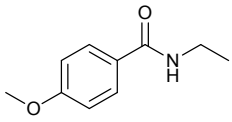
55.4, 39.8, 31.7, 21.4, 20.1, 13.7; IR (KBr) ν 3291, 3068, 2860, 2928, 1638, 1596, 1541; HRMS (ESI-TOF) calcd for $C_{13}H_{19}NNaO_2$ (M + Na^+) 244.1313, found 244.1310.

4-Methoxy-N-methylbenzamide (4a)[7]



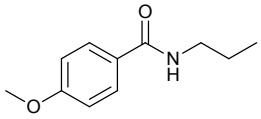
Yellow solid; yield: 78%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.73 (d, *J* = 7.7 Hz, 2H), 6.88 (d, *J* = 7.7 Hz, 2H), 6.39 (bs, 1H), 3.81 (s, 3H), 2.96 (d, *J* = 2.9 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.8, 162.1, 128.6, 127.0, 113.7, 55.3, 26.7.

N-Ethyl-4-methoxybenzamide (4b)[8]



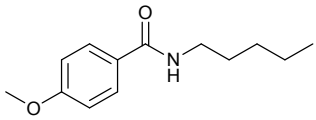
Yellow solid; yield: 79%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.72 (d, *J* = 8.7 Hz, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 6.47 (bs, 1H), 3.79 (s, 3H), 3.44–3.39 (m, 2H), 1.19 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.1, 161.9, 128.6, 126.9, 113.5, 55.2, 34.8, 14.8.

4-Methoxy-N-propylbenzamide (4c)[9]



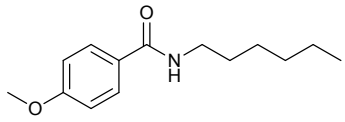
Yellow solid; yield: 73%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.72 (d, *J* = 8.9 Hz, 2H), 6.89 (d, *J* = 8.9 Hz, 2H), 6.22 (bs, 1H), 3.82 (s, 3H), 3.39–3.36 (m, 2H), 1.68–1.54 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.1, 162.0, 128.6, 127.1, 113.6, 55.3, 41.7, 22.9, 11.4.

4-Methoxy-N-pentylbenzamide (4e)[3]



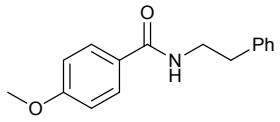
Yellow solid; yield: 59%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.73 (d, *J* = 8.6 Hz, 2H), 6.90 (d, *J* = 8.6 Hz, 2H), 6.15 (bs, 1H), 3.83 (s, 3H), 3.44–3.39 (m, 2H), 1.65–1.50 (m, 2H), 1.35–1.30 (m, 4H), 0.91–0.88 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.0, 161.9, 128.6, 127.1, 113.6, 55.3, 40.0, 29.3, 29.1, 22.3, 13.9.

N-Hexyl-4-methoxybenzamide (4f)[10]



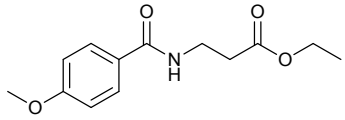
Yellow solid; yield: 70%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.73 (d, *J* = 8.6 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.30 (bs, 1H), 3.81 (s, 3H), 3.42–3.37 (m, 2H), 1.60–1.53 (m, 2H), 1.38–1.22 (m, 6H), 0.88–0.85 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.0, 161.9, 128.6, 127.1, 113.6, 55.3, 40.0, 31.5, 29.6, 26.6, 22.5, 14.0.

4-Methoxy-N-phenethylbenzamide (4g)[11]



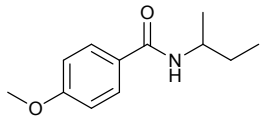
White solid; yield: 70%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.68 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 6.7 Hz, 2H), 7.24–7.16 (m, 3H), 6.85 (d, *J* = 8.3 Hz, 2H), 6.49 (bs, 1H), 3.79 (s, 3H), 3.66–3.65 (m, 2H), 2.90–2.89 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 167.0, 162.0, 139.0, 128.7, 128.6, 128.5, 127.0, 126.4, 113.6, 55.2, 41.0, 35.7.

Ethyl 3-(4-methoxybenzamido)propanoate (4h)[12]



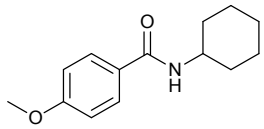
Yellow solid; yield: 89%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.75 (d, *J* = 7.4 Hz, 2H), 7.02 (bs, 1H), 6.89 (d, *J* = 7.4 Hz, 2H), 4.16–4.15 (m, 2H), 3.82 (s, 3H), 3.70–3.69 (m, 2H), 2.66–2.60 (m, 2H), 1.26 (s, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 172.7 , 166.9, 162.1, 128.6, 126.7, 113.6, 60.6, 55.2, 35.3, 34.0, 14.0.

N-(sec-butyl)-4-methoxybenzamide (4i)[13]



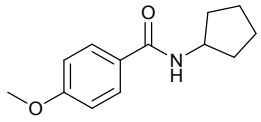
Yellow solid; yield: 83%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.72 (d, *J* = 8.7 Hz, 2H), 6.89 (d, *J* = 8.7 Hz, 2H), 5.92 (bs, 1H), 4.18–4.01 (m, 1H), 3.82 (s, 3H), 1.65–1.47 (m, 2H), 1.20 (d, *J* = 6.5 Hz, 3H), 0.94 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 166.4, 161.9, 128.5, 127.3, 113.6, 55.3, 46.9, 29.7, 20.5, 10.4.

N-Cyclohexyl-4-methoxybenzamide (4j)[14]



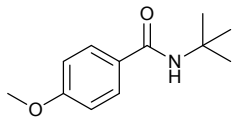
White solid; yield: 60%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.71 (d, *J* = 8.6 Hz, 2H), 6.90 (d, *J* = 8.6 Hz, 2H), 5.95 (bs, 1H), 3.96–3.91 (m, 1H), 3.83 (s, 3H), 2.02–2.00 (m, 2H), 1.76–1.72 (m, 2H), 1.45–1,36 (m, 2H), 1.24–1.17 (m, 4H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 166.1, 161.9, 128.6, 127.3, 113.6, 55.3, 48.5, 33.2, 25.5, 24.9.

N-Cyclopentyl-4-methoxybenzamide (4k)[8]



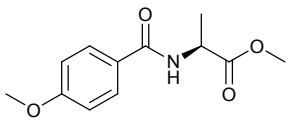
White solid; yield: 59%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.71 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.6 Hz, 2H), 6.04 (bs, 1H), 4.40–4.33 (m, 1H), 3.83 (s, 3H), 2.09–2.05 (m, 2H), 1.77–1.57 (m, 4H), 1.49–1.45 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 166.6, 161.9, 128.6, 127.2, 113.6, 55.3, 51.6, 33.2, 23.8.

N-(tert-butyl)-4-methoxybenzamide (4l)[15]

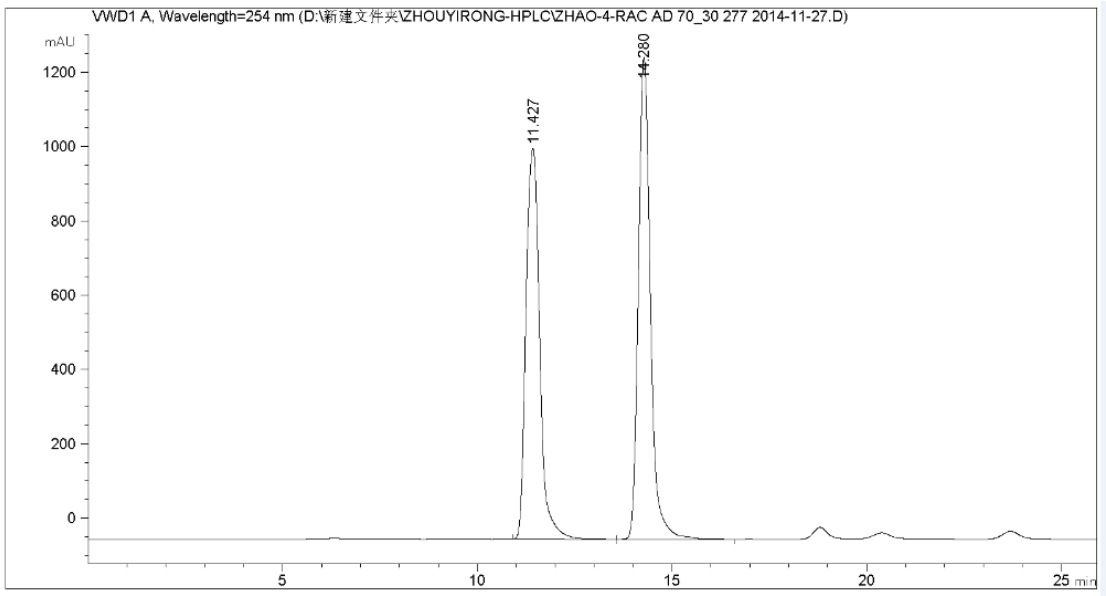


White solid; yield: 85%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.68 (d, *J* = 8.2 Hz, 2H), 6.89 (d, *J* = 8.3 Hz, 2H), 5.89 (bs, 1H), 3.83 (s, 3H), 1.45 (s, 9H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 166.4, 161.8, 128.4, 128.1, 113.5, 55.3, 51.4, 28.9.

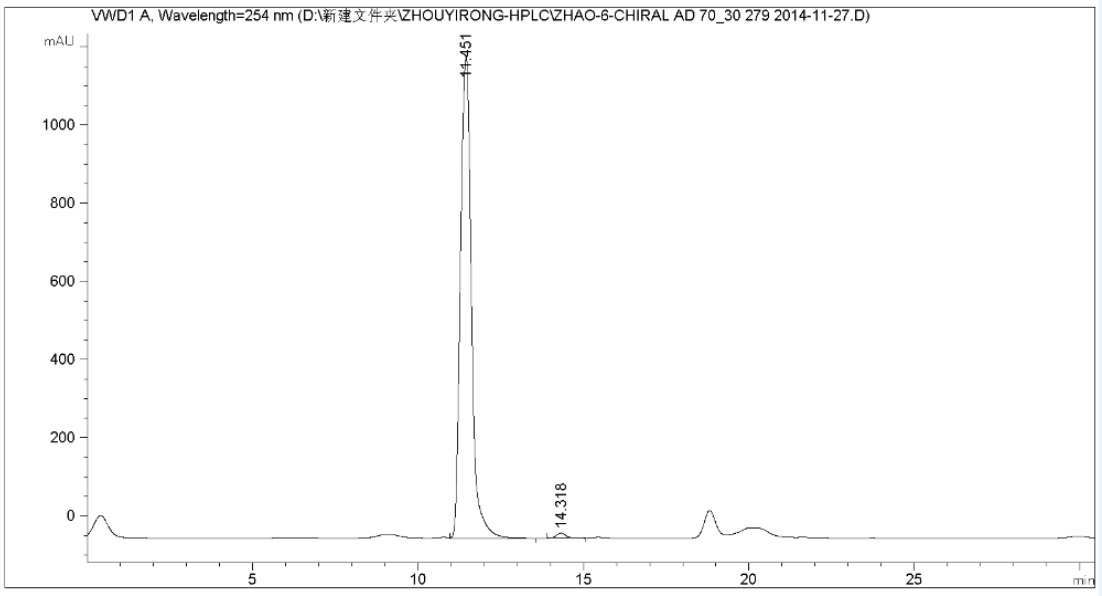
(S)-Methyl 2-(4-methoxybenzamido)propanoate (4m)[16]



White solid; yield: 58%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.77 (d, *J* = 8.6 Hz, 2H), 6.91 (d, *J* = 8.5 Hz, 2H), 6.69 (bs, 1H), 4.81–4.75 (m, 1H), 3.84 (s, 3H), 3.78 (s, 3H), 1.51 (d, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 173.9, 166.3, 162.4, 128.9, 126.1, 113.8, 55.4, 52.5, 48.4, 18.7; **HPLC** (Chiral AD, n-hexane/isopropanol = 70:30, flow rate = 0.5 mL/min) tR = 11.427 , tR = 14.280 min, Ee = 98.3%.

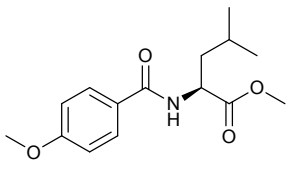


(rac- HPLC spectrum)



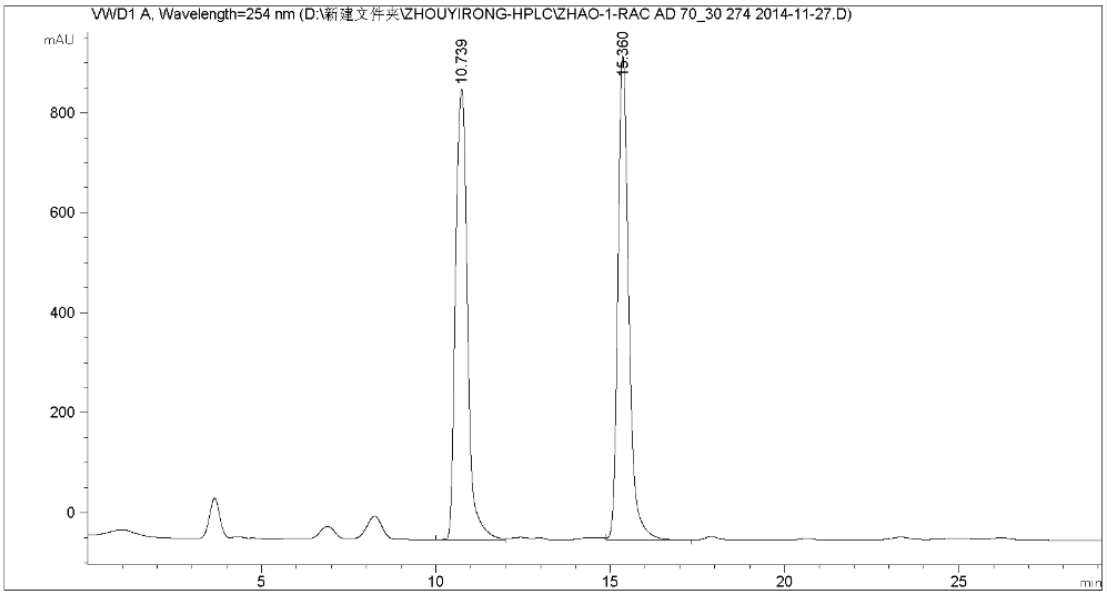
(4m HPLC spectrum)

(S)-Methyl 2-(4-methoxybenzamido)-4-methylpentanoate (4n)[17]

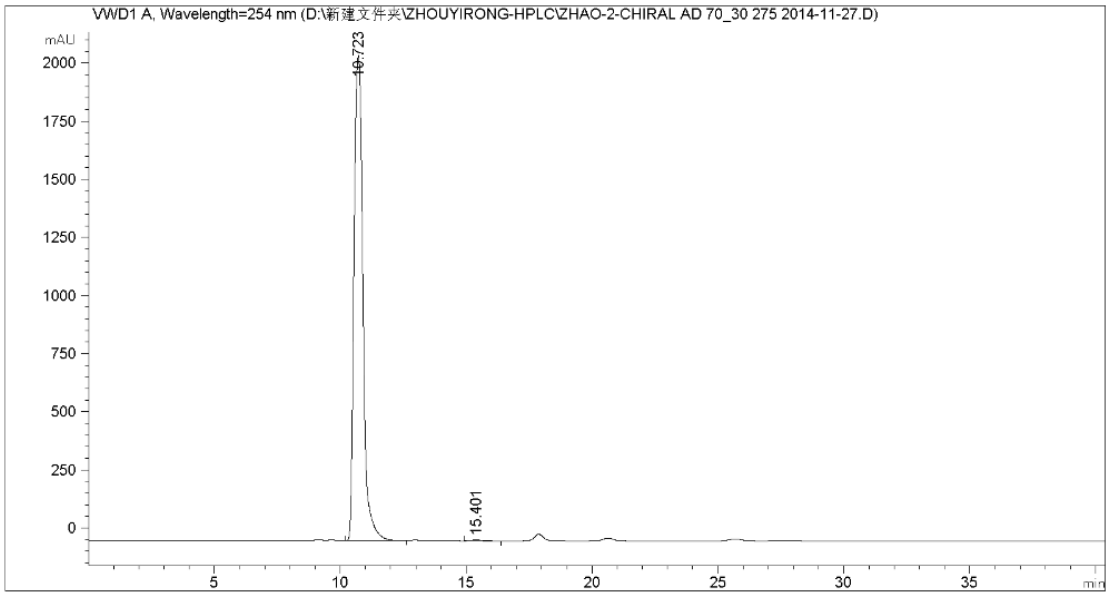


White solid; yield: 59%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.78–7.70 (m, 2H), 6.92–6.85 (m, 2H), 6.53 (d, *J* = 8.0 Hz, 1H), 4.87–4.81 (m, 1H), 3.83 (s, 3H), 3.75 (s, 3H), 1.77–1.68 (m, 2H), 1.66–1.60 (m, 1H), 0.98–0.95 (m, 6H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 173.9, 166.7, 162.4, 128.9, 126.1, 113.7, 55.4, 52.4, 51.1, 41.8, 25.0, 22.9, 22.1; **HPLC** (Chiral AD, n-hexane/isopropanol =70:30, flow rate =

0.5 mL/min) tR = 10.739 , tR = 15.360 min, Ee = 99.6%.

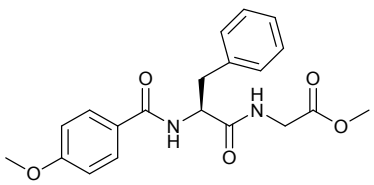


(rac- HPLC spectrum)



(4n HPLC spectrum)

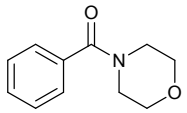
Methyl 2-(2-(4-methoxybenzamido)-3-phenylpropanamido)acetate (4o)



White solid; yield: 40%; **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.68 (d, *J* = 8.4 Hz, 2H), 7.30–7.14 (m, 7H),

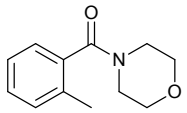
6.84 (d, *J* = 8.3 Hz, 2H), 5.04–4.99 (m, 1H), 3.95 (d, *J* = 5.1 Hz, 2H), 3.81 (s, 3H), 3.67 (s, 3H), 3.29–3.18 (m, 2H); **^{13}C NMR (100 MHz, $CDCl_3$):** δ 171.8, 170.0, 167.1, 162.4, 136.7, 129.4, 129.0, 128.6, 126.9, 125.9, 113.7, 55.4, 54.6, 52.3, 41.2, 38.1; IR (KBr) ν 3311, 3085, 1754, 1658, 1625, 1606, 1572, 980; HRMS (ESI-TOF) calcd for $C_{20}H_{22}N_2NaO_5$ (M + Na^+) 393.1426, found 393.1452.

Morpholino(phenyl)methanone (6a)[1]



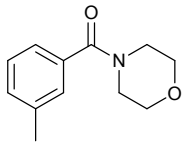
Yellow liquid; yield: 74%; **^{1}H NMR (400 MHz, CDCl3)** δ 7.43–7.37 (m, 5H), 4.29–2.83 (m, 8H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 170.4 , 135.3, 129.8, 128.5, 127.0, 66.8(2C), 48.2, 42.4.

Morpholino(o-tolyl)methanone (6b)[7]



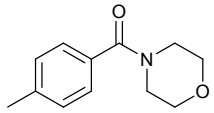
Colourless liquid; yield: 55%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.30–7.26 (m, 1H), 7.23–7.20 (m, 2H), 7.17–7.15 (m, 1H), 3.83–3.77 (m, 4H), 3.61–3.54 (m, 2H), 3.25–3.23 (m, 2H), 2.32 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 170.1, 135.6, 134.1, 130.4, 129.0, 125.9, 125.8, 66.9, 47.2, 41.9, 19.0.

Morpholino(m-tolyl)methanone (6c)[18]



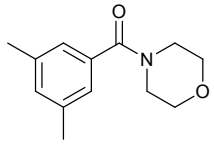
Yellow liquid; yield: 78%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.31–7.16 (m, 4H), 3.76–3.43 (m, 8H), 2.38 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 170.6, 138.5, 135.3, 130.5, 128.3, 127.6, 123.9, 66.9, 48.1, 42.5, 21.3.

Morpholin-4-yl-p-toly-methanone (6d)[1]



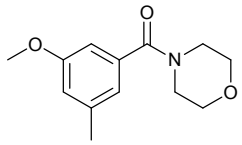
Colourless liquid; yield: 66%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.29 (d, J = 7.8 Hz, 2H), 7.20 (d, J = 7.8 Hz, 2H), 3.66–3.52 (m, 8H), 2.36 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 170.5, 140.0, 132.2, 129.0, 127.1, 66.8, 48.0, 42.5, 21.3.

(3,5-Dimethylphenyl)(morpholino)methanone (6e)[1]



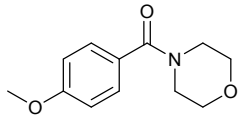
White solid; yield: 60%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.04 (s, 1H), 6.99 (s, 2H), 3.69–3.45 (m, 8H), 2.33 (s, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 170.8, 138.2, 135.3, 131.3, 124.6, 66.9, 48.2, 42.2, 21.2.

(3-Methoxy-5-methylphenyl)(morpholino)methanone (6f)



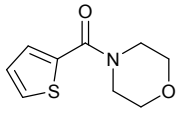
Yellow liquid; yield: 61%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 6.76 (s, 2H), 6.71 (s, 1H), 3.79 (s, 3H), 3.61 – 3.44 (m, 8H), 2.33 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 170.3, 159.6, 140.0, 136.4, 119.8, 116.2, 109.4, 67.0, 55.3, 48.2, 42.5, 21.4; IR (KBr) ν 3050, 2857, 1625, 1606, 1591, 1003, 960; HRMS (ESI-TOF) calcd for $C_{13}H_{18}NO_3$ (M + H^+) 236.1287, found 236.1283.

(4-Methoxyphenyl)(morpholino)methanone (6g)[8]



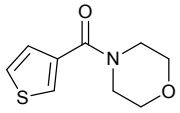
Yellow liquid; yield: 81%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.38 (d, *J* = 8.6 Hz, 2H), 6.91 (d, *J* = 7.9 Hz, 1H), 3.82 (s, 3H), 3.70 – 3.61 (m, 8H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 170.3, 160.8, 129.1, 127.1, 113.6, 66.8, 55.2, 48.1, 43.0.

Morpholino(thiophen-2-yl)methanone (6h)[18]



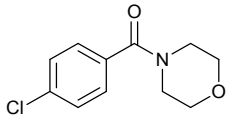
Yellow liquid; yield: 63%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.47 (d, *J* = 5.0 Hz, 1H), 7.30 – 7.28 (m, 1H), 7.08–7.04 (m, 1H), 3.77–3.73 (m, 8H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 163.6, 136.5, 128.9, 128.8, 126.7, 66.8, 45.7.

Morpholino(thiophen-3-yl)methanone (6i)[19]



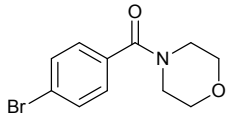
Yellow liquid; yield: 73%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.51 (dd, *J* = 2.8, 1.1 Hz, 1H), 7.33 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.16 (dd, *J* = 5.0, 1.0 Hz, 1H), 3.67 (s, 8H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 165.7, 135.7, 126.8, 126.6, 126.1, 66.7, 47.8, 42.6.

(4-chlorophenyl)(morpholino)methanone (6j)[2]



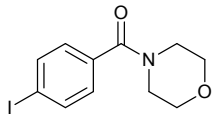
Yellow solid; yield: 71%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.39–7.33 (m, 4H), 3.90–3.20 (m, 8H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 169.4, 136.0, 133.6, 128.9, 128.7, 66.8, 48.3, 42.6.

(4-Bromophenyl)(morpholino)methanone (6k)[18]



Yellow solid; yield: 67%; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.56 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 2H), 3.74 – 3.44 (m, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 169.2, 134.0, 131.9, 128.7, 124.1, 66.7, 48.1, 42.5.

(4-Iodophenyl)(morpholino)methanone (6l)[20]



White solid；yield：84%；**^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.77 –7.74 (m, 2H), 7.15–7.12 (m, 2H), 3.69–3.44 (m, 8H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 169.5, 137.7, 134.7, 128.8, 96.1, 66.8, 48.1, 42.5.

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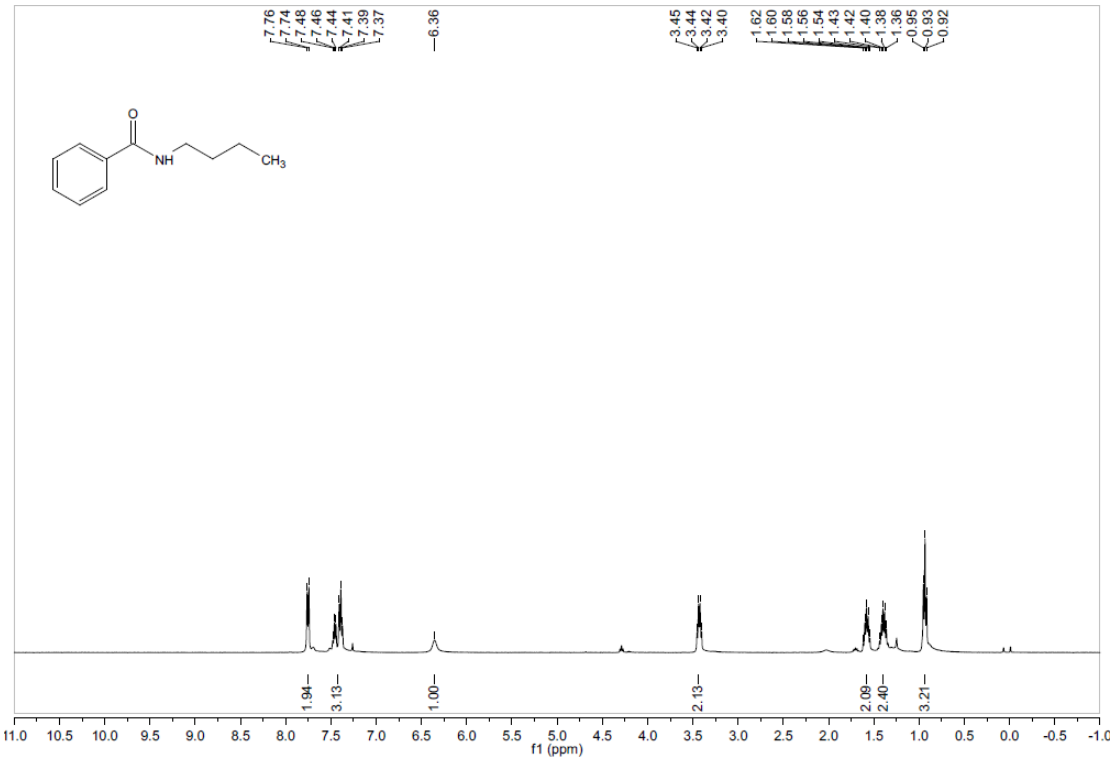
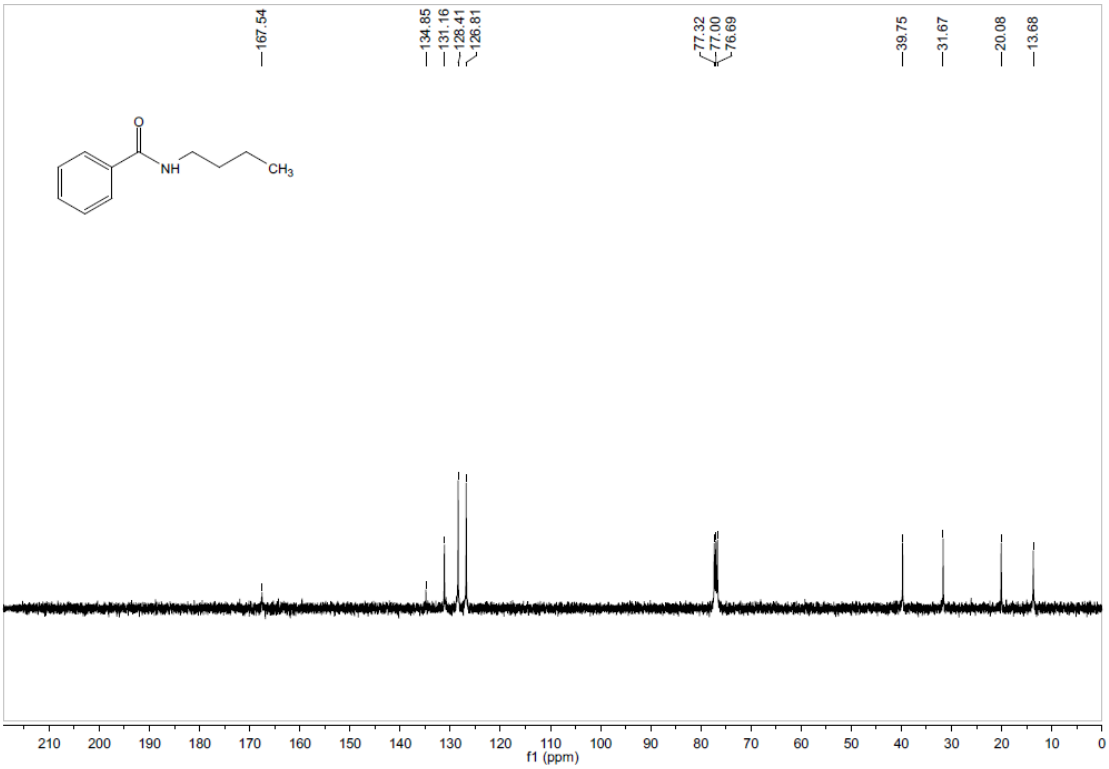
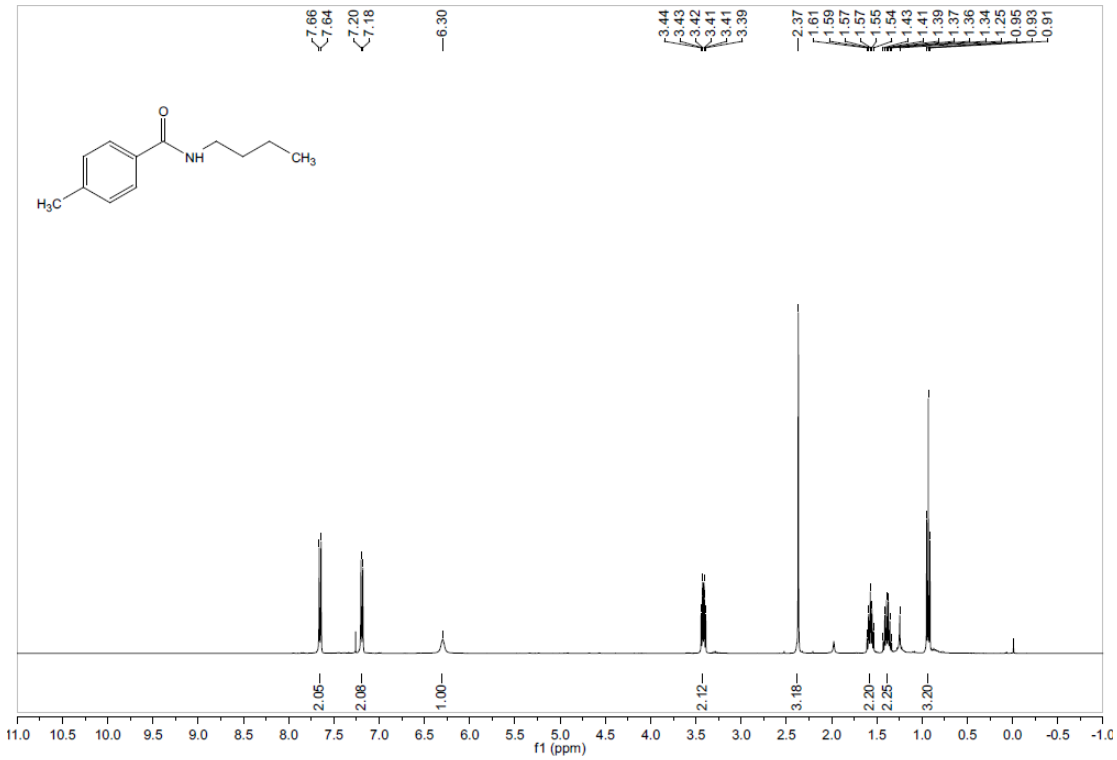
References：

1. R. Vanjari, T. Guntreddi and K. N. Singh, *Org. Lett.,* 2013, **15**, 4908.
2. M. Pilo, A. Porcheddu and L. D. Luca, *Org. Biomol. Chem.,* 2013, **11**, 8241–8246.
3. X. F. Wu, M. Sharif, A. Pews-Davtyan, P. Langer, K. Ayub, and M. Beller, *Eur. J. Org. Chem.,* 2013, 2783–2787.
4. A. R. Prosser, J. E. Banning, M. Rubina, and M. Rubin, *Org. Lett.*, 2010, **12**, 3968–3971.
5. L. Wang, C. Zhong, P. Xue, and E. Q. Fu, *J. Org. Chem.,* 2011, **76**, 4874–4883.
6. X. Y. Wu and M. Larhed, *Org. Lett.*, 2005, **7**, 3327–3329.
7. Y. Jo, J. Ju, J. Choe, K. H. Song, and S. Lee, *J. Org. Chem*., 2009, **74**, 6358–6361.
8. P. Baburajan, K.P. Elango, *Tetrahedron Lett.,* 2014, **55**, 1006–1010.
9. V. de la Fuente , C. Godard, C. Claver, S. Castillon, *Adv. Synth. Catal*., 2012, **354**, 1971–1979.
10. P. Hermange, A. T. Lindhardt, R. H. Taaning, K. Bjerglund, D. Lupp, and T. Skrydstrup, *J. Am. Chem. Soc*., 2011, **133**, 6061–6071.
11. D. Xing, X. F. Xu and L. P. Yang, *Synthesis*., 2009,3399–3404.
12. Y. C. Jeong, Z. Bikadi, E. Hazai, M. G. Moloney, *ChemMedChem*., 2014, **9**,1826–1837.
13. C. Jamieson, M. S. Congreve, D. F. Emiabata-Smith, S. V. Ley, *Synlett*., 2000, 1603–1607.
14. G. Pelletier, W. S. Bechara, and A. B. Charette, *J. Am. Chem. Soc.*, 2010, **132**, 12817–12819.
15. H. Jiang , B. Liu , Y. Li , A. Wang , and H. Huang, *Org. Lett.*, 2011, **13**, 1028–1031.
16. K. D. Ginzel, P. Brungs, E. Steckhan, *Tetrahedron*, 1989, **45**, 1691–1701.
17. H. W. Li, X. C. Lu, J. G.Yang, K. Jiang, X. X. Fang, Y. Q. Wu, *Gaodeng Xuexiao Huaxue Xuebao*, 2009, **30**, 716–719.
18. W. Fang, Q. Deng, M. Xu, and T. Tu, *Org. Lett.*, 2013, **15**, 3678–3681.
19. S. D. Friis, T. Skrydstrup, and S. L. Buchwald, *Org. Lett.,* 2014, **16**, 4296−4299.
20. E. Brachet, J. D. Brion, M. Alami, S. Messaoudi, *Chem.–Eur. J.*, 2013, **19**, 15276–15280.

Copies of ^{1}H-NMR & ^{13}C-NMR spectrum



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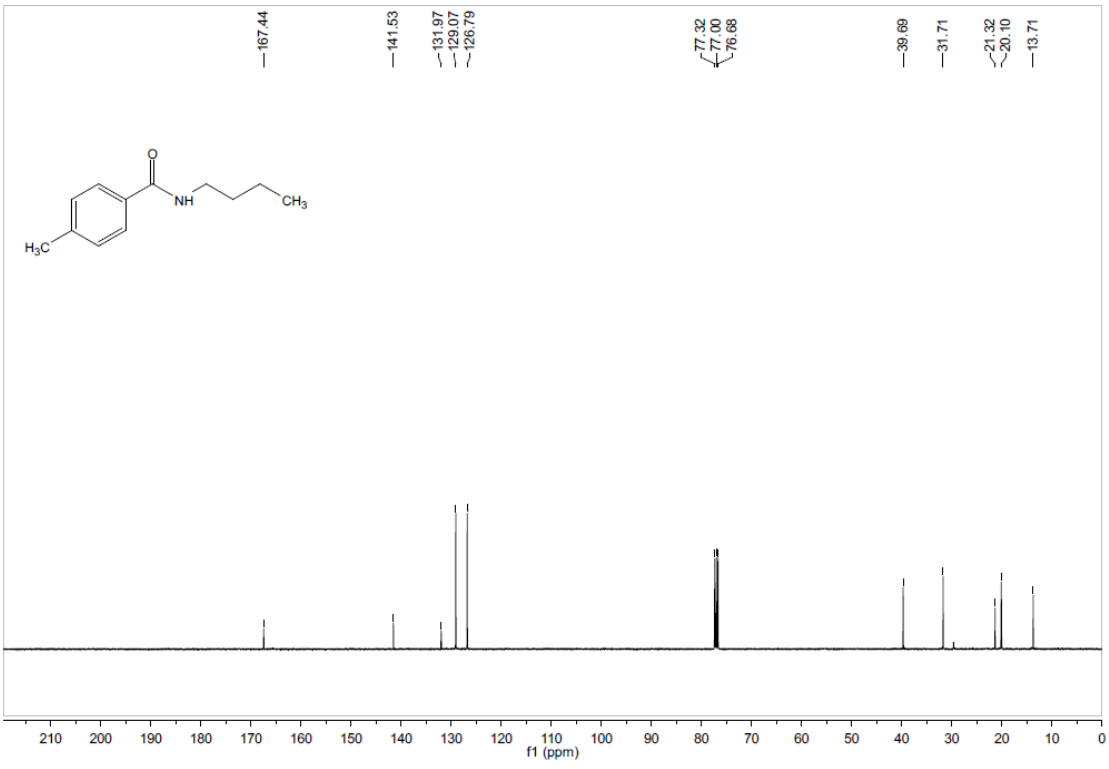
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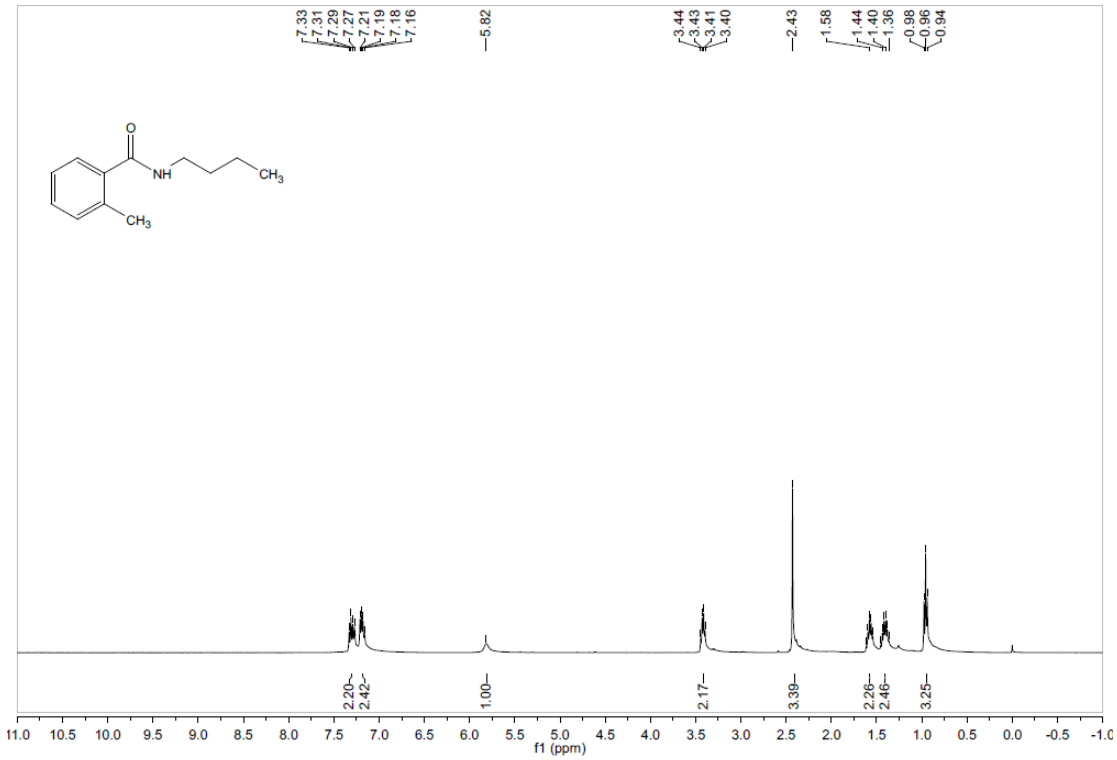
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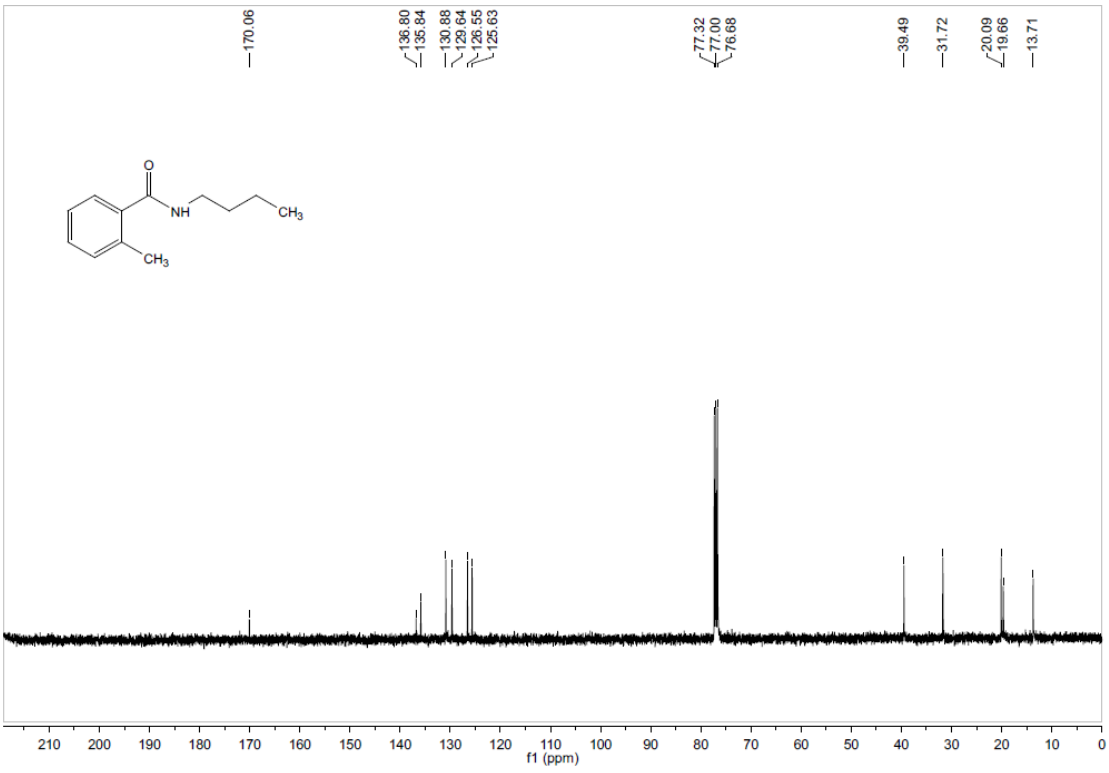
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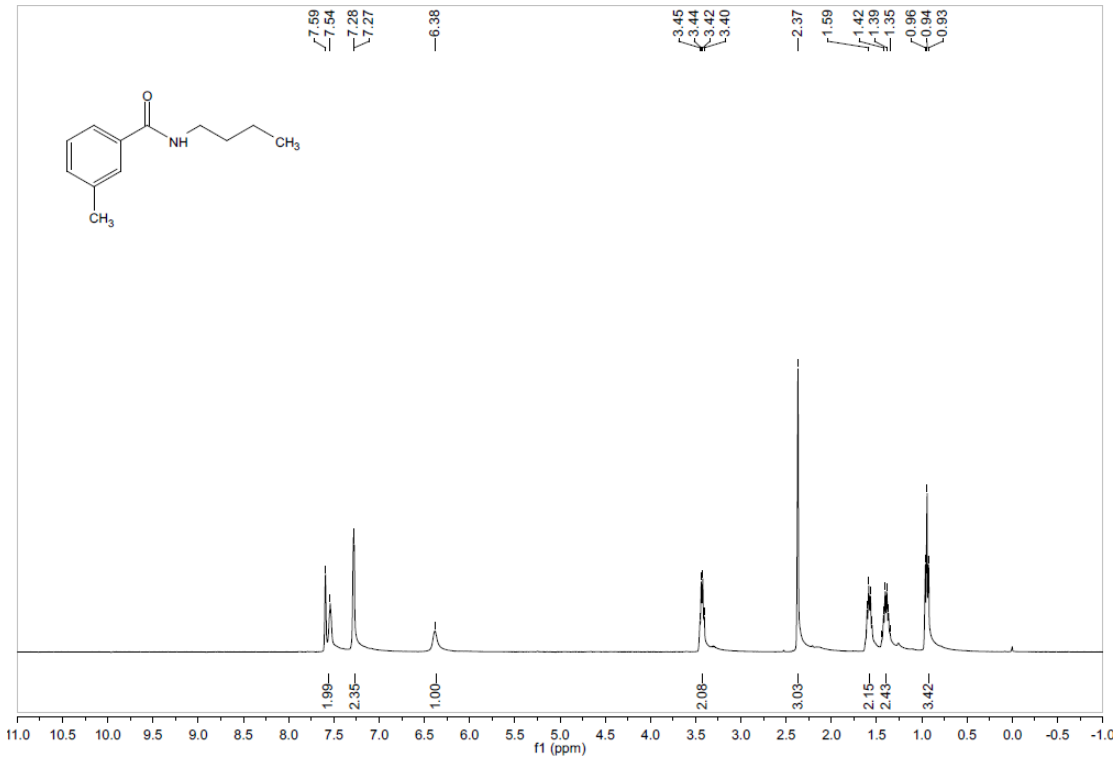
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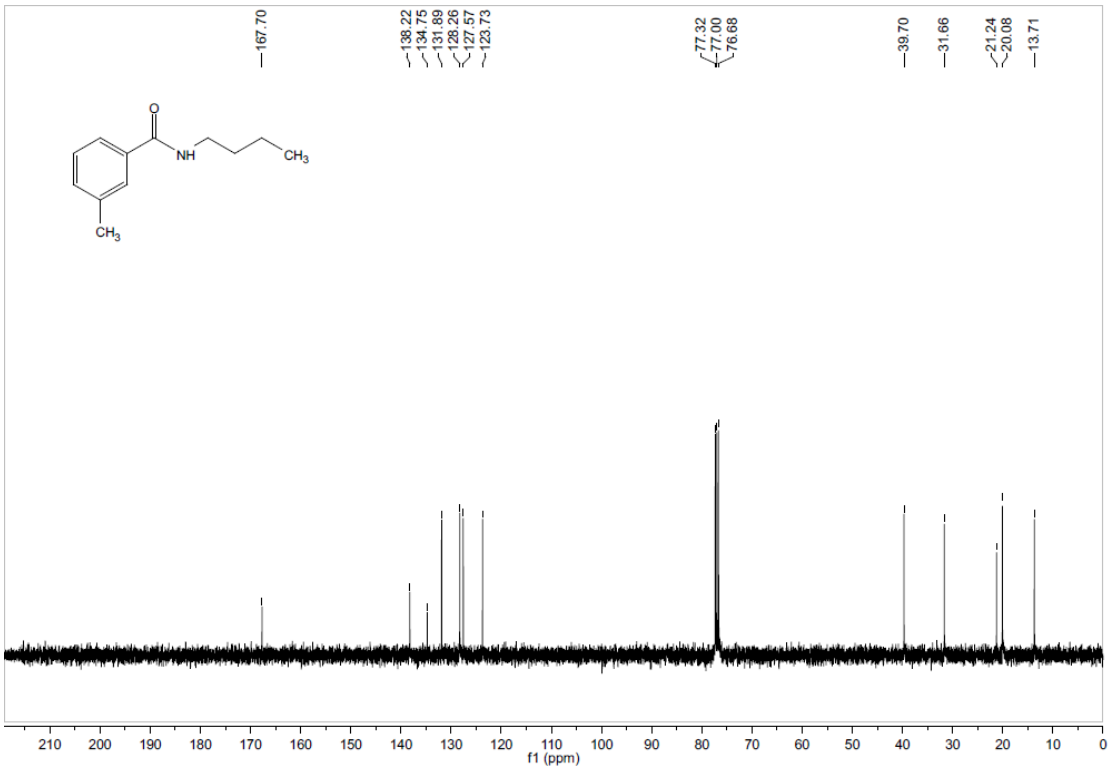
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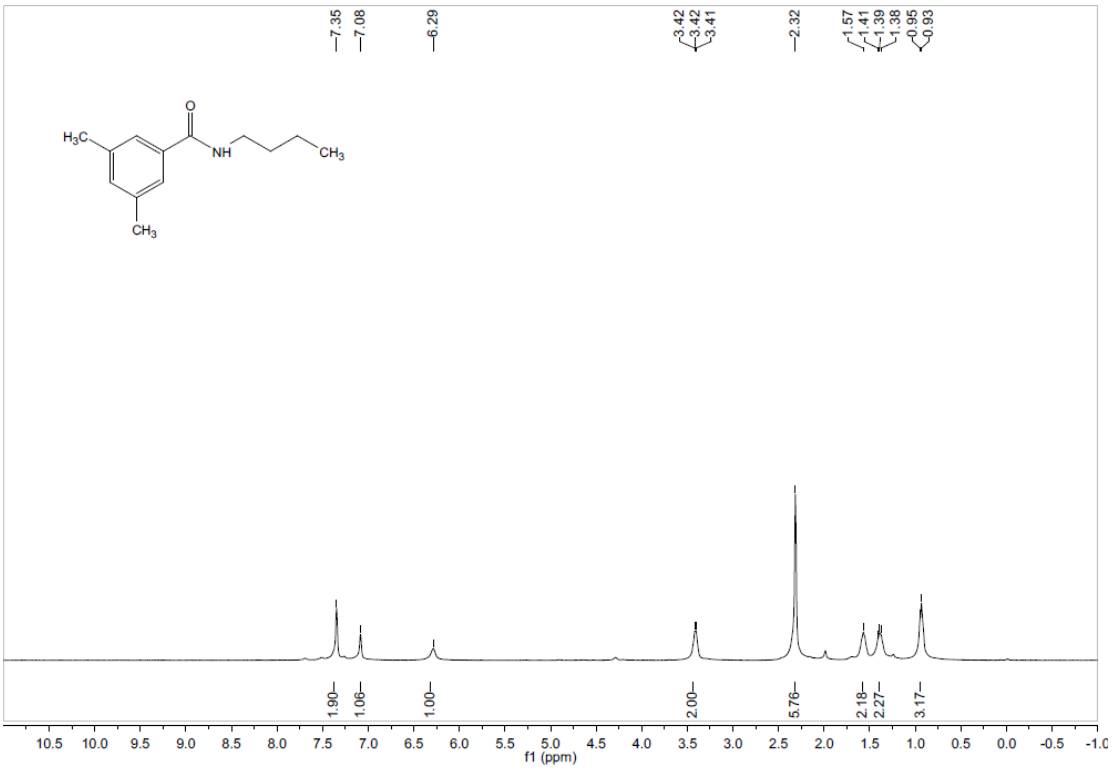
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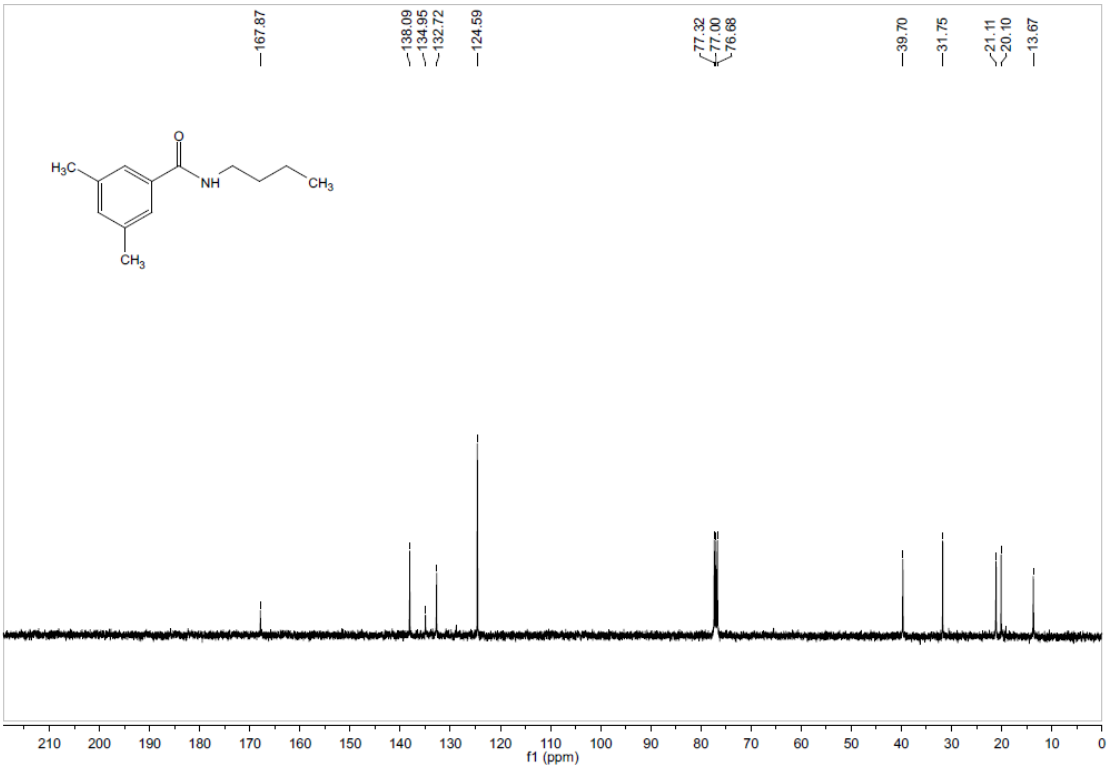
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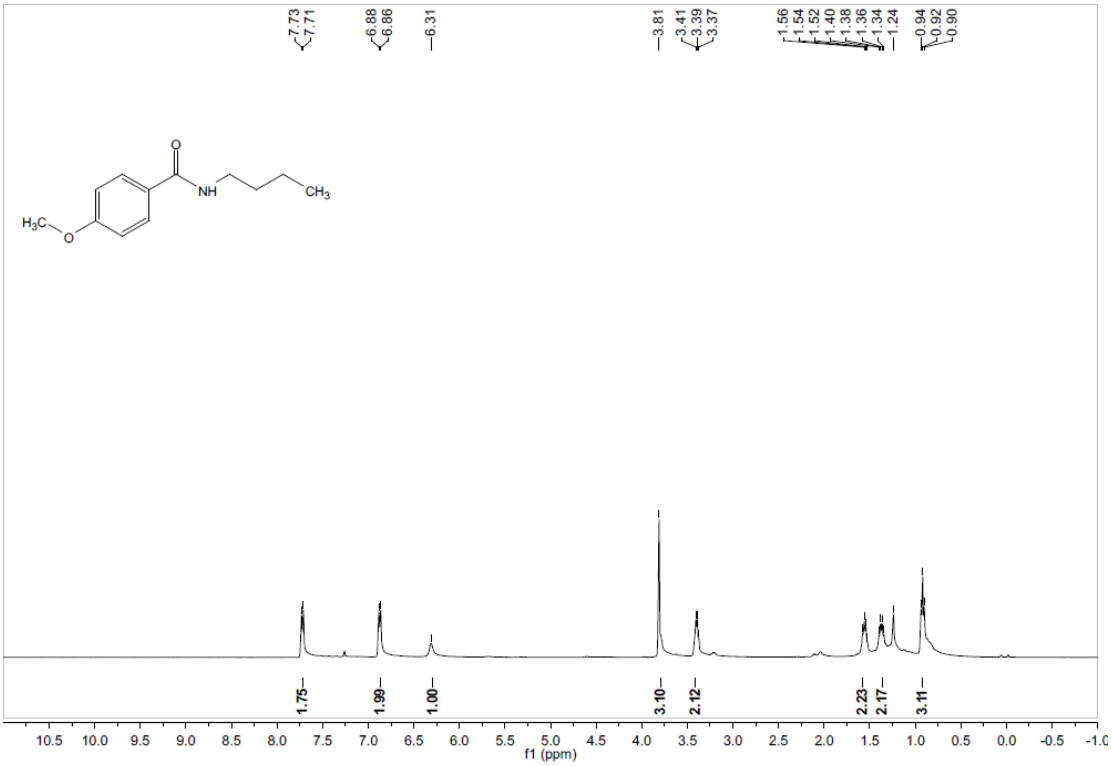
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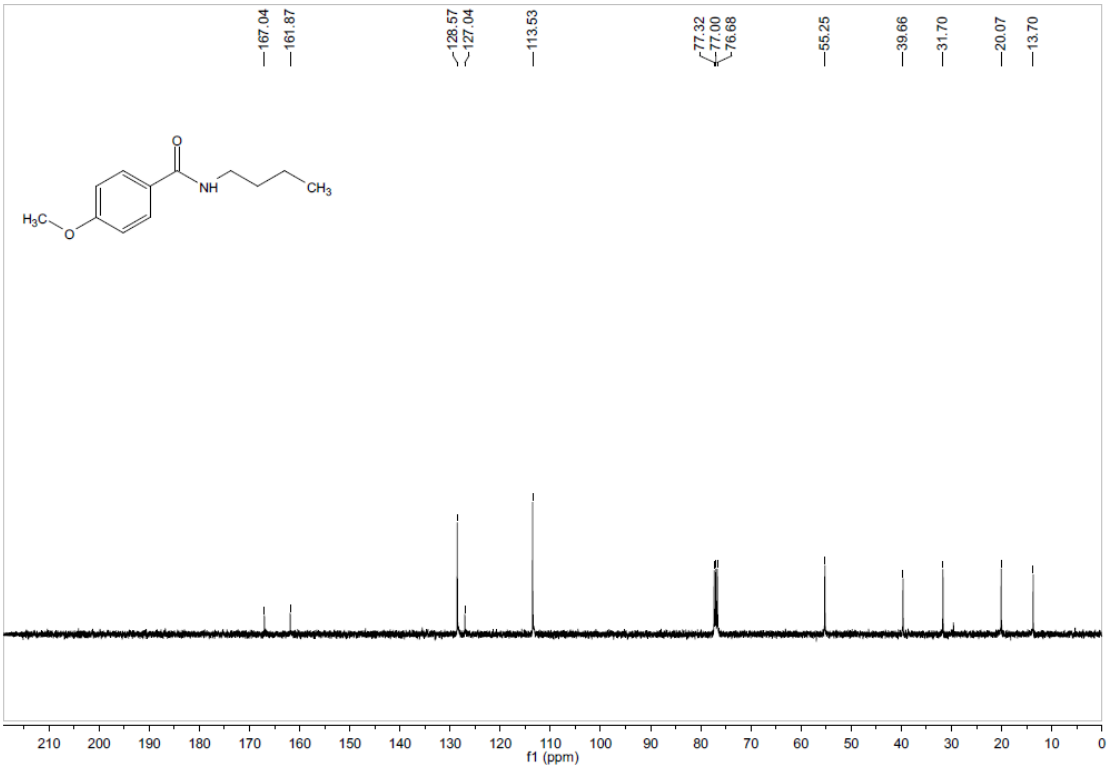
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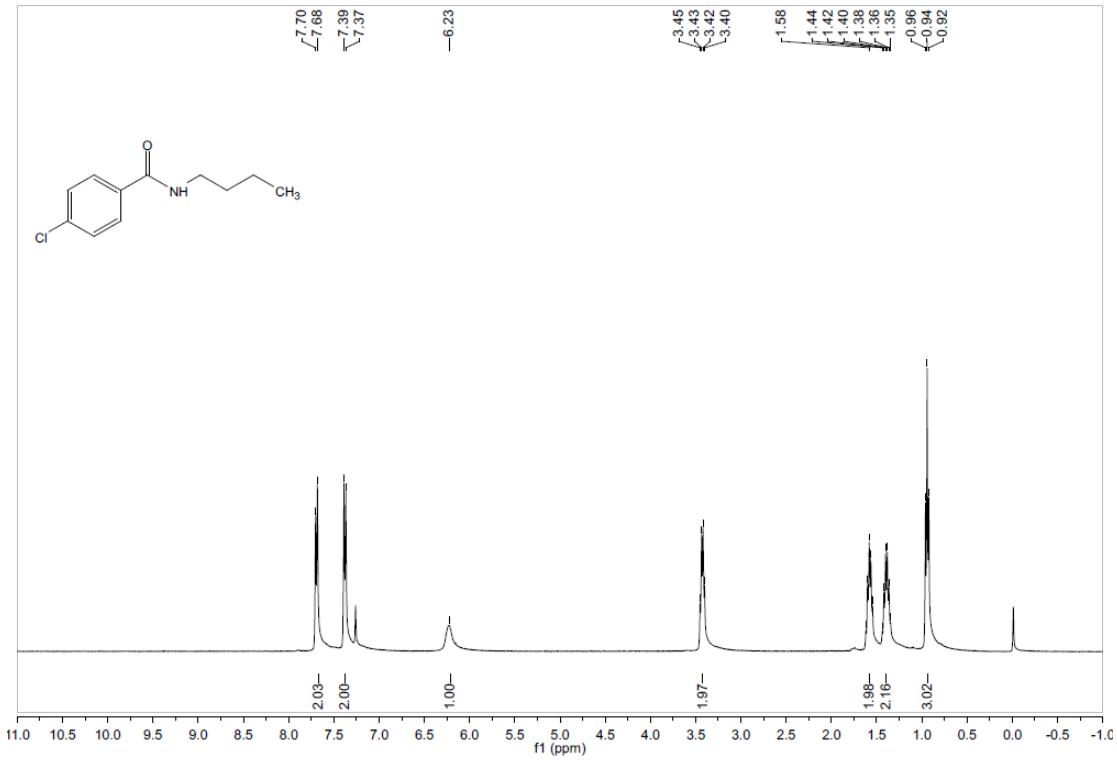
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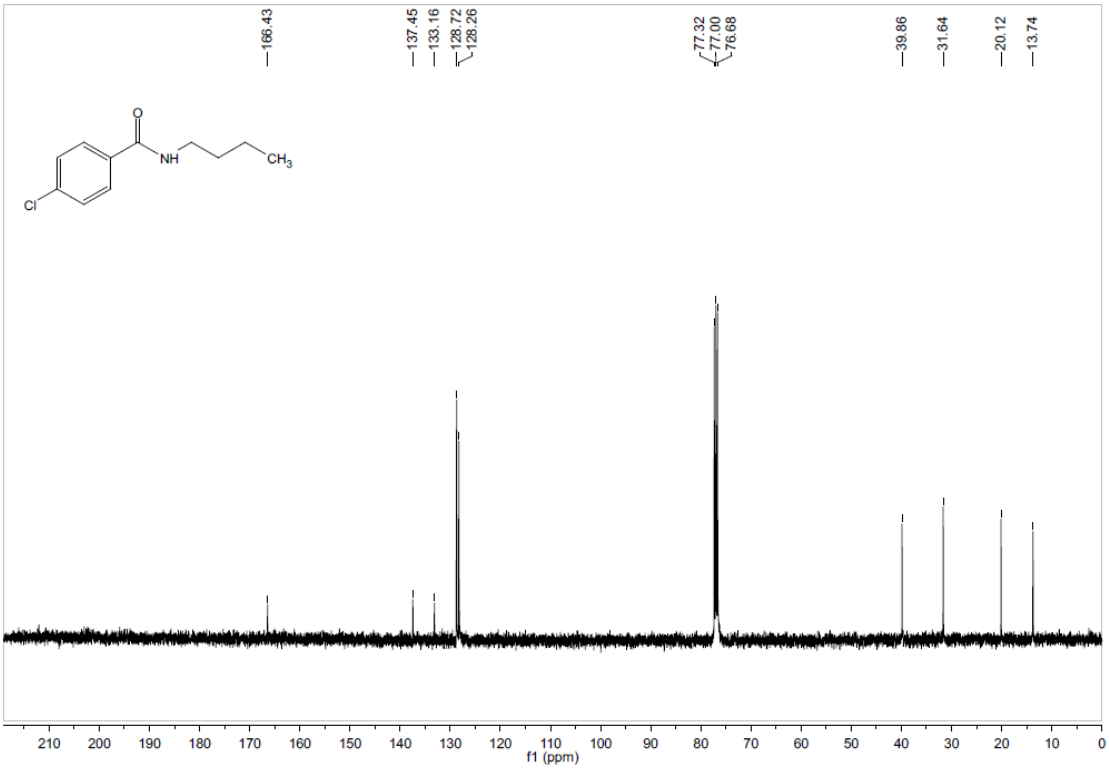
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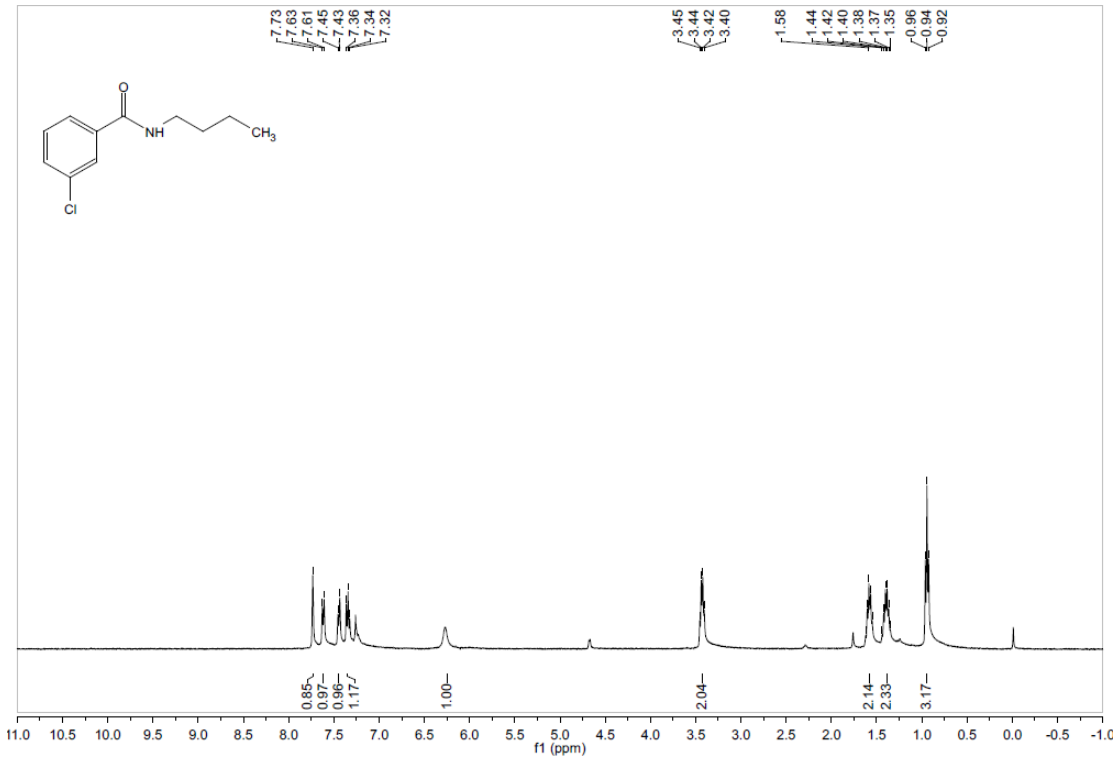
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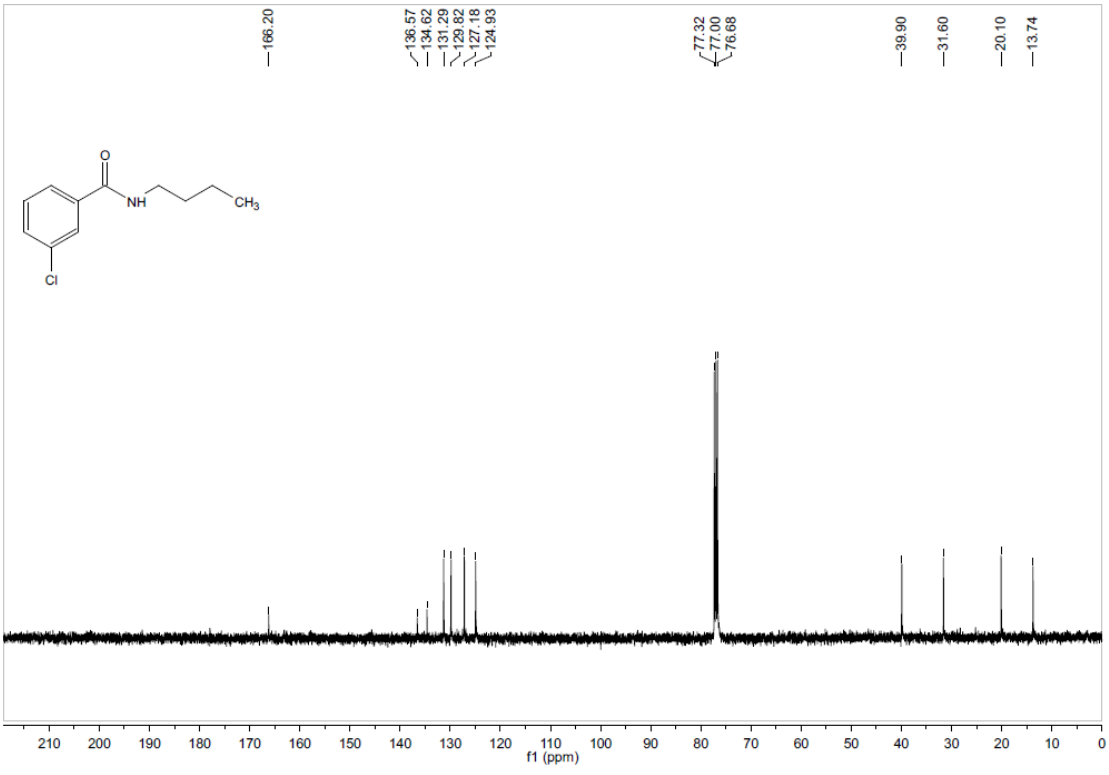
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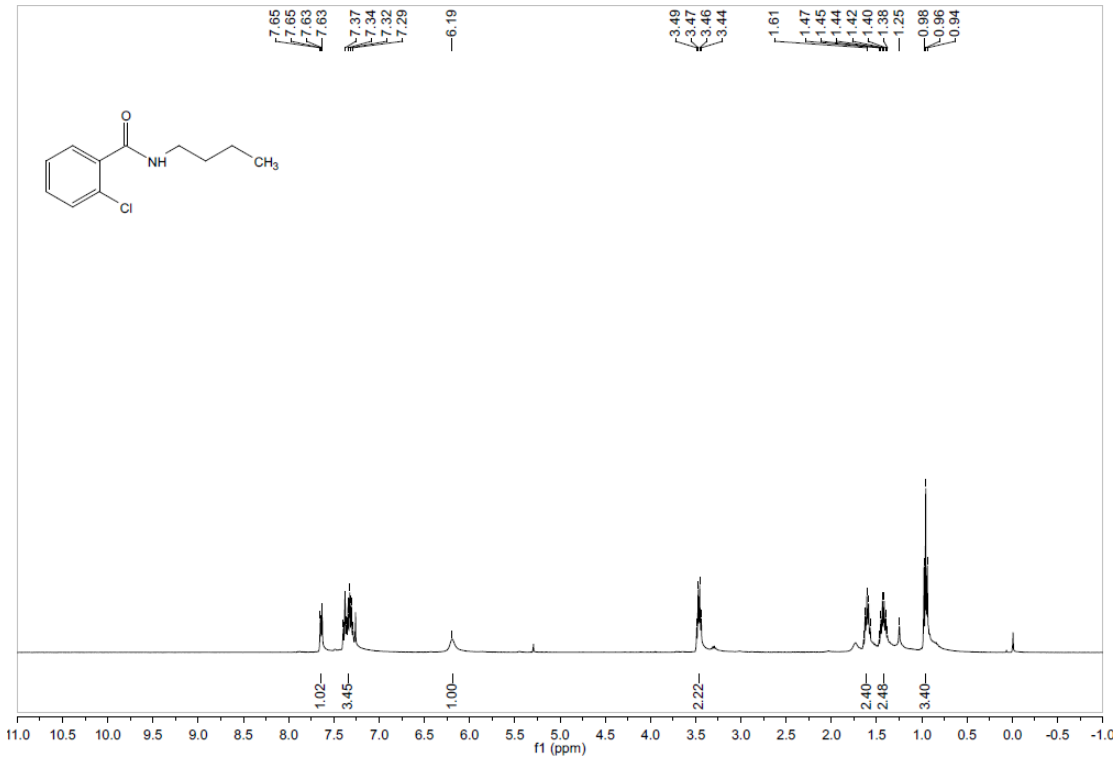
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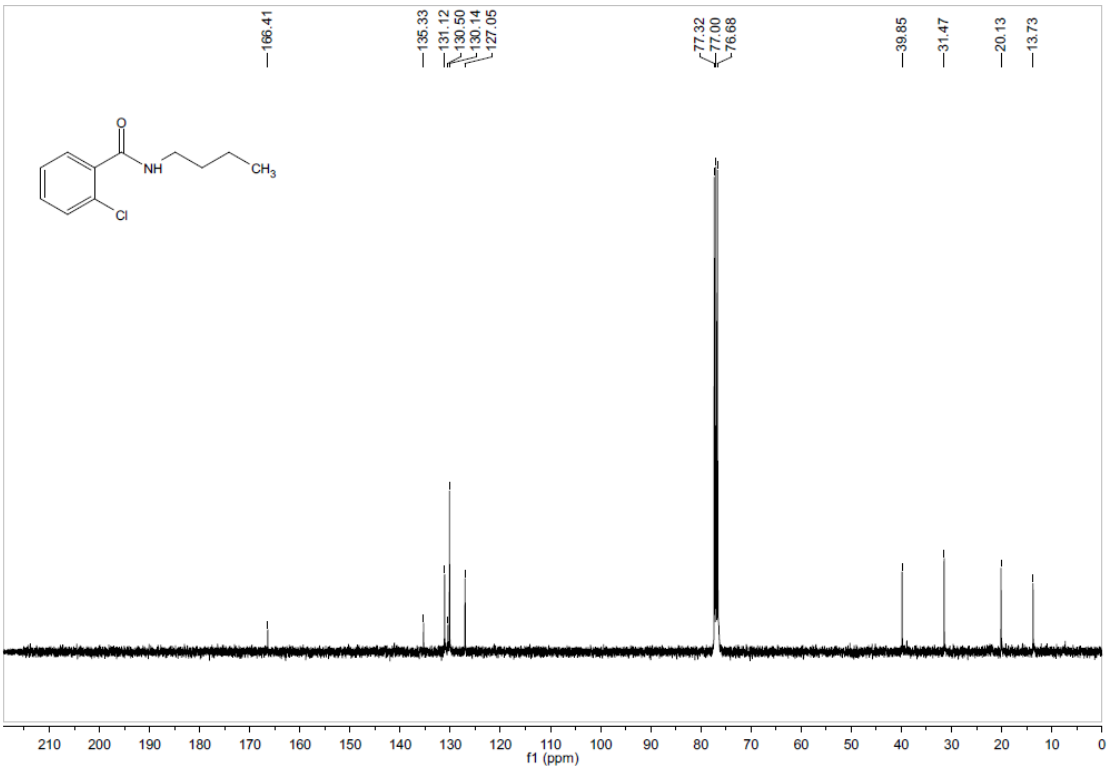
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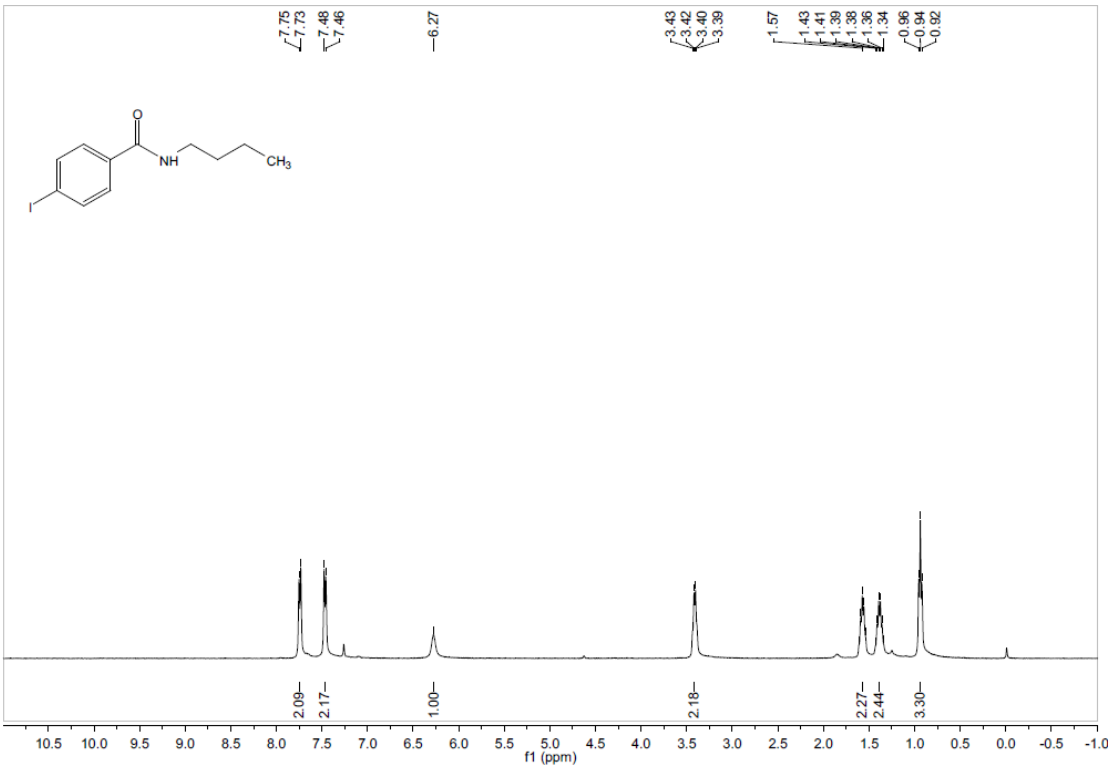
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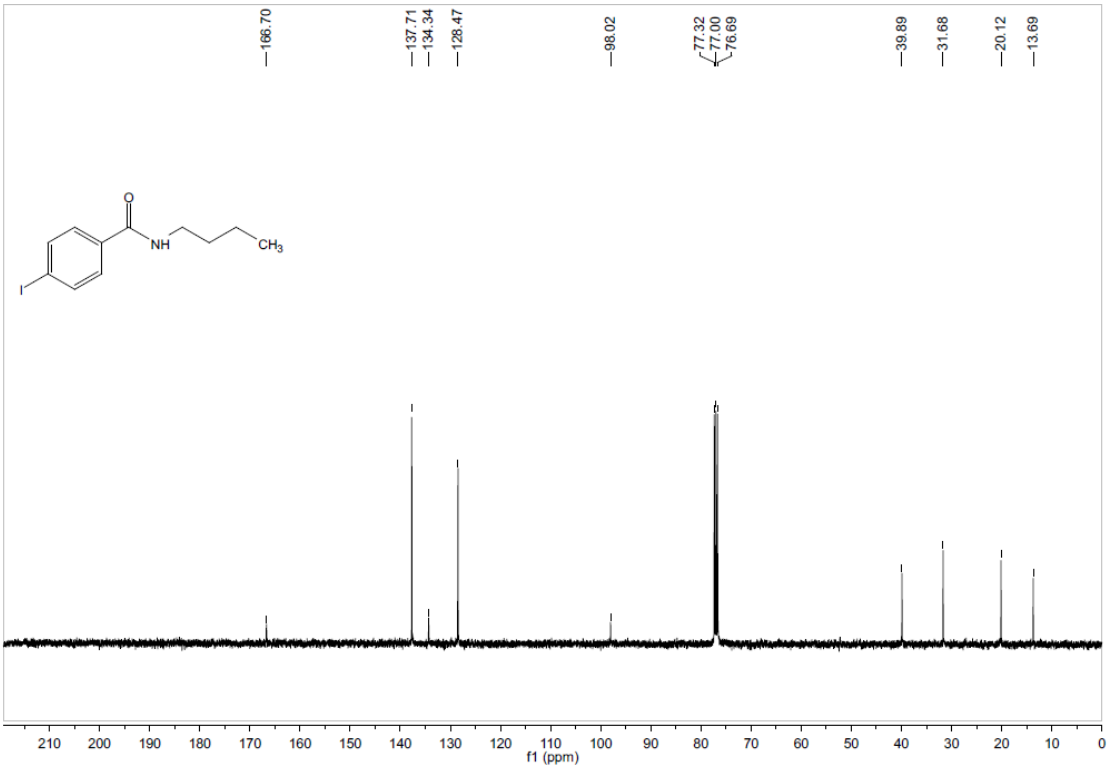
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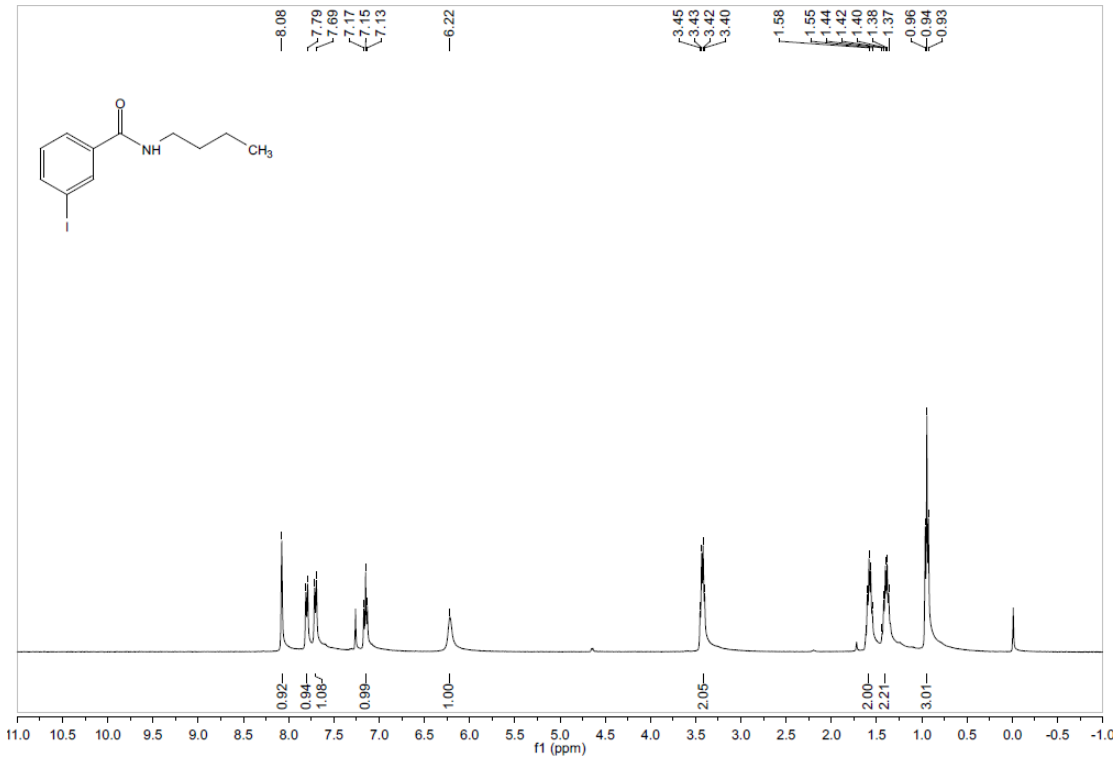
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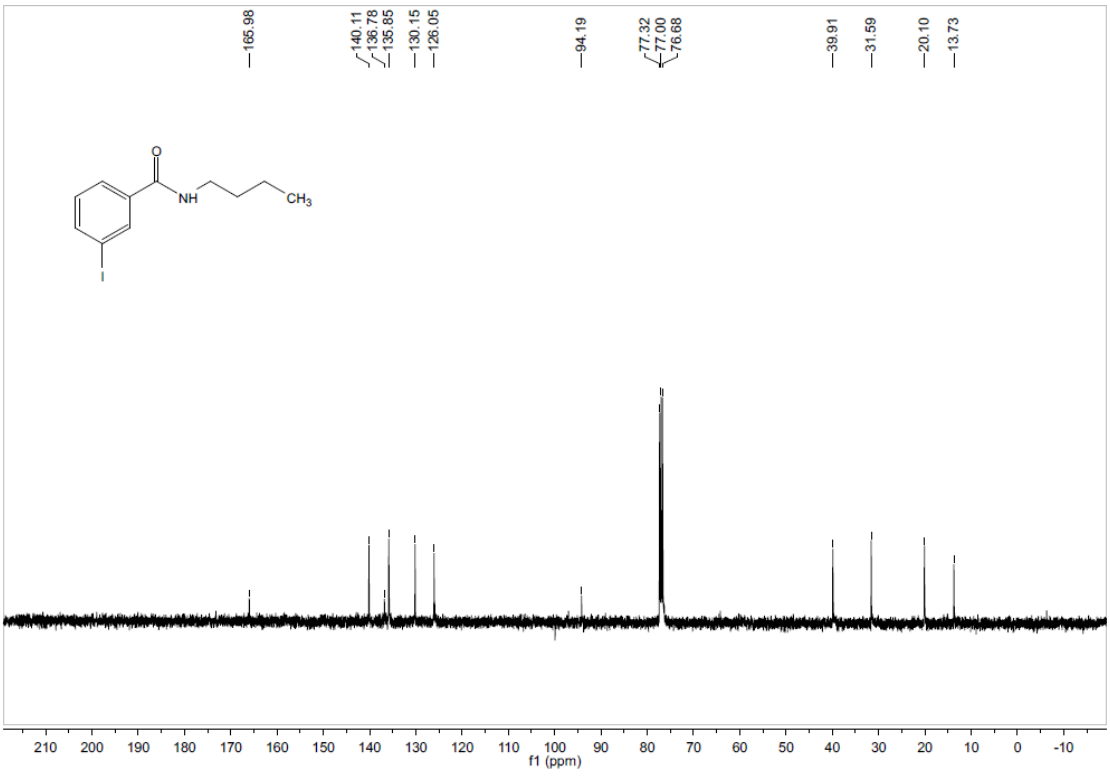
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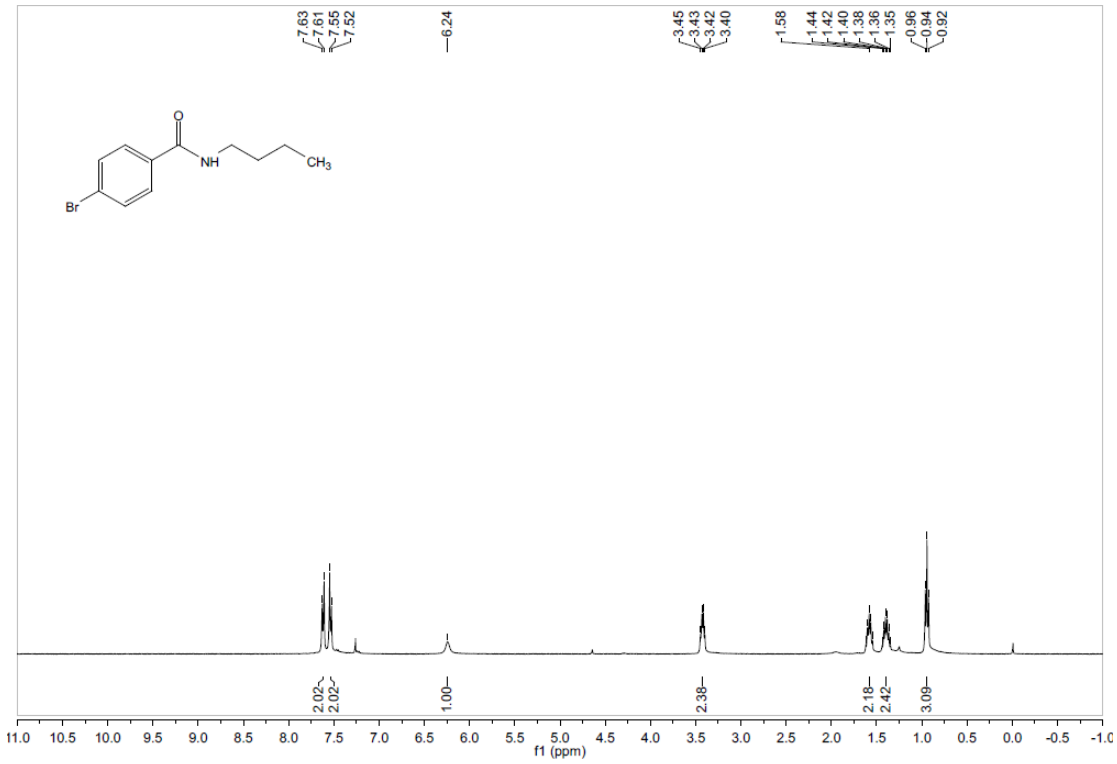
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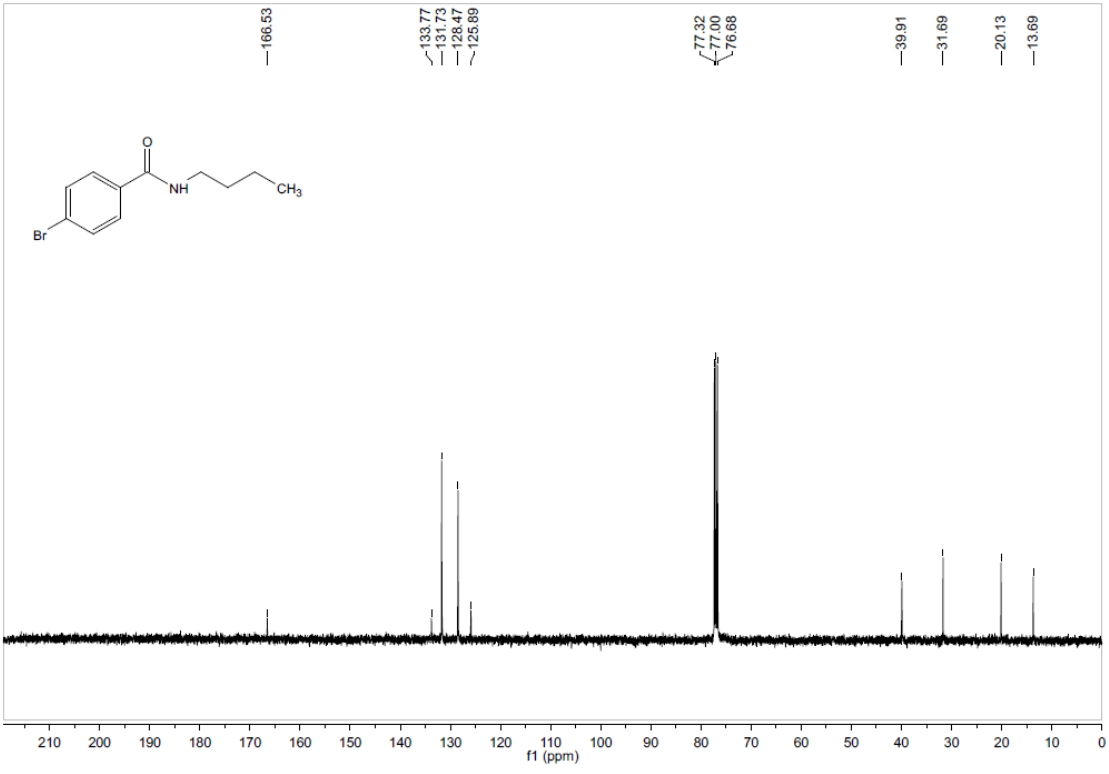
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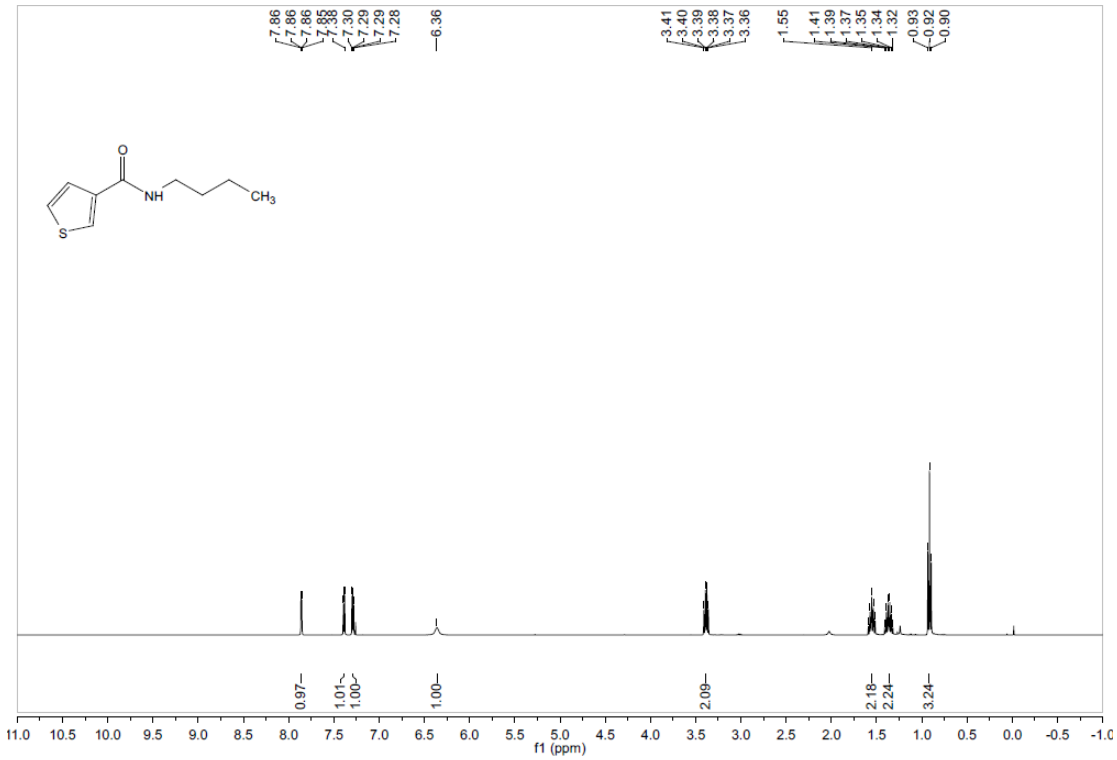
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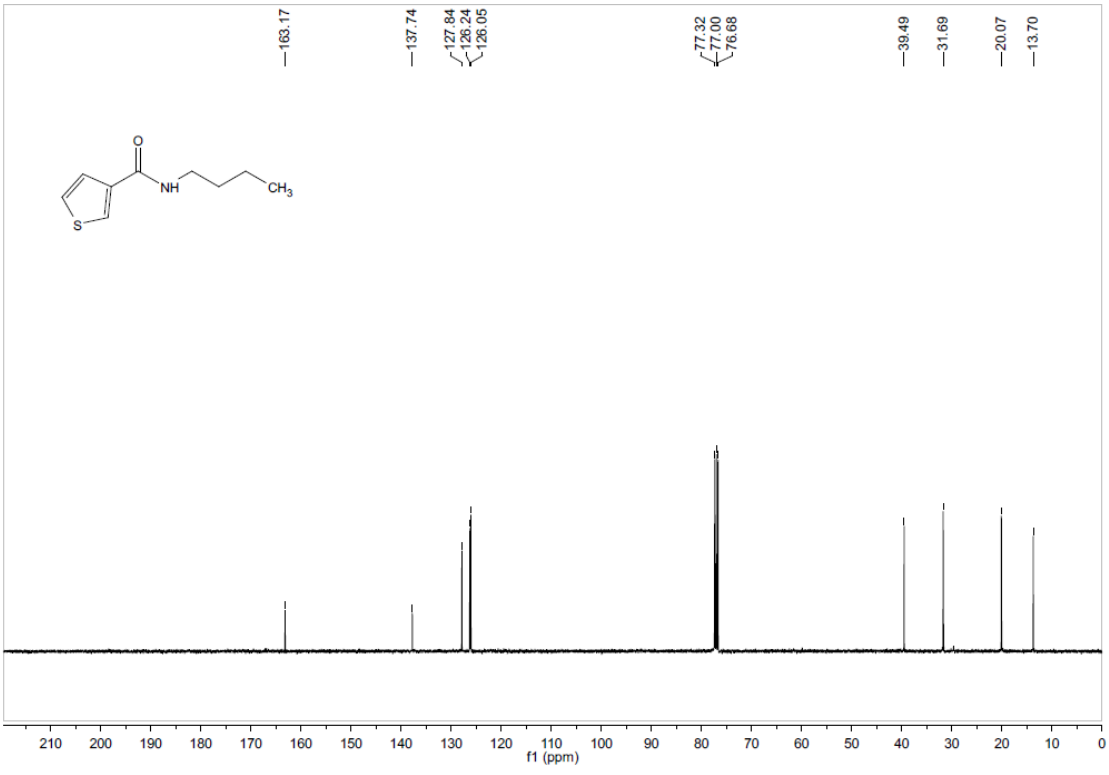
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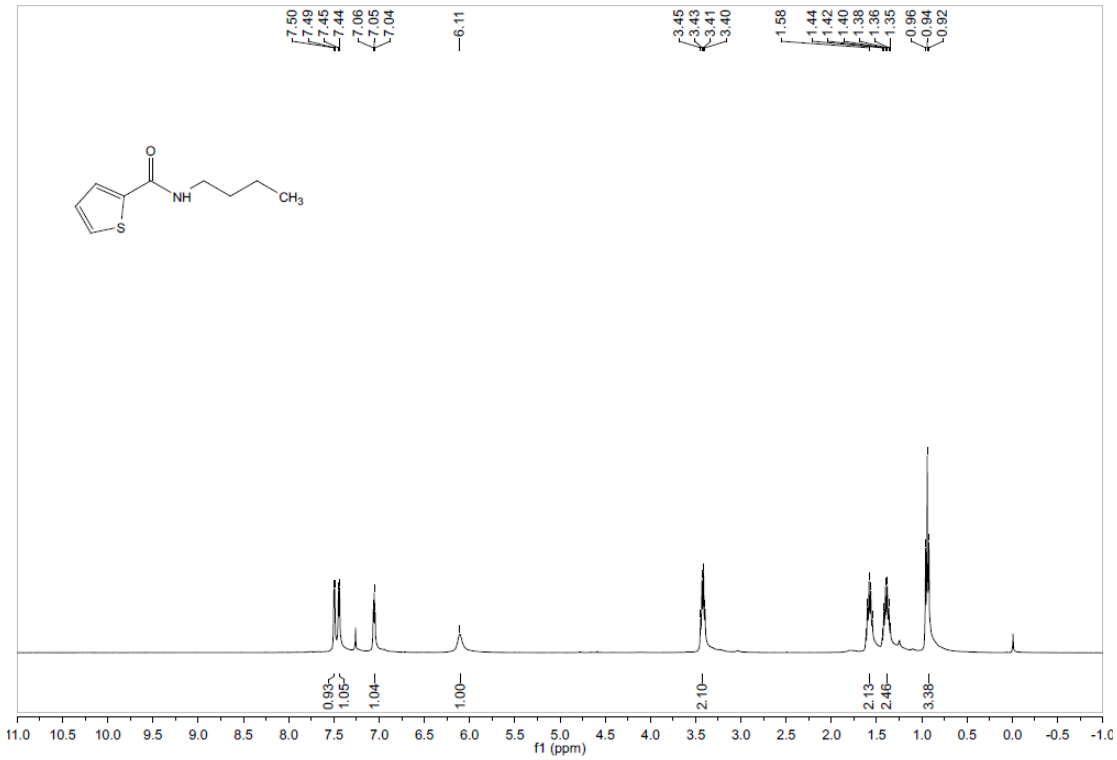
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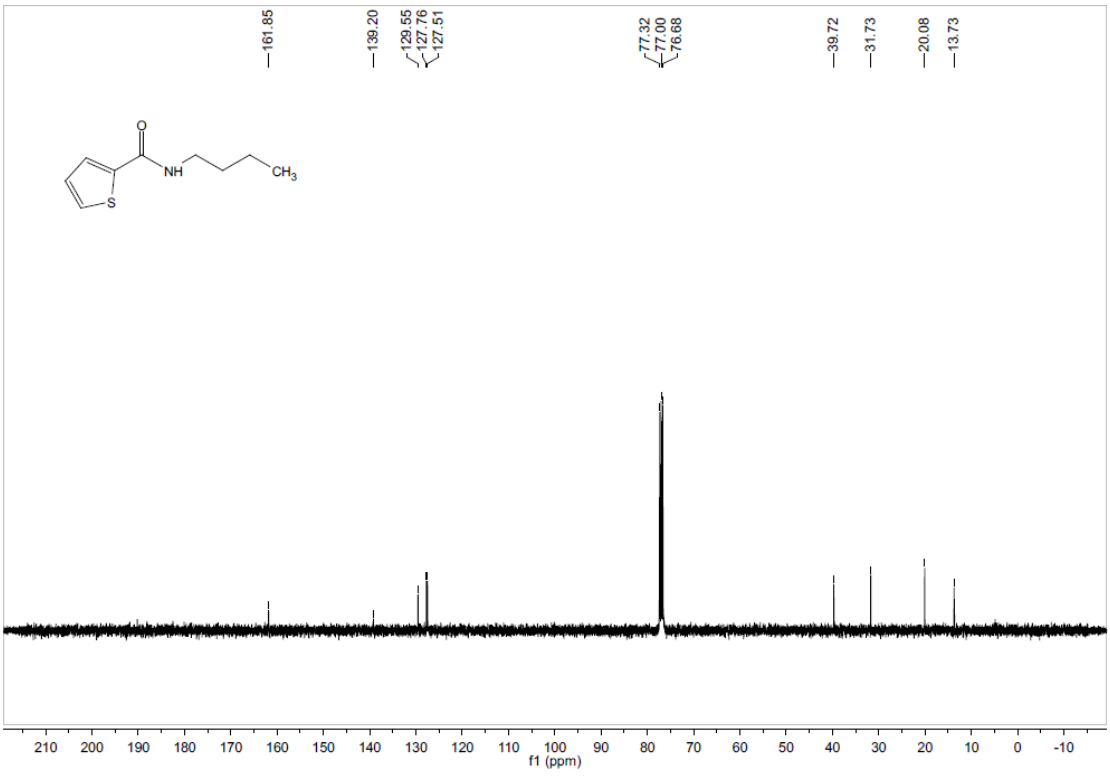
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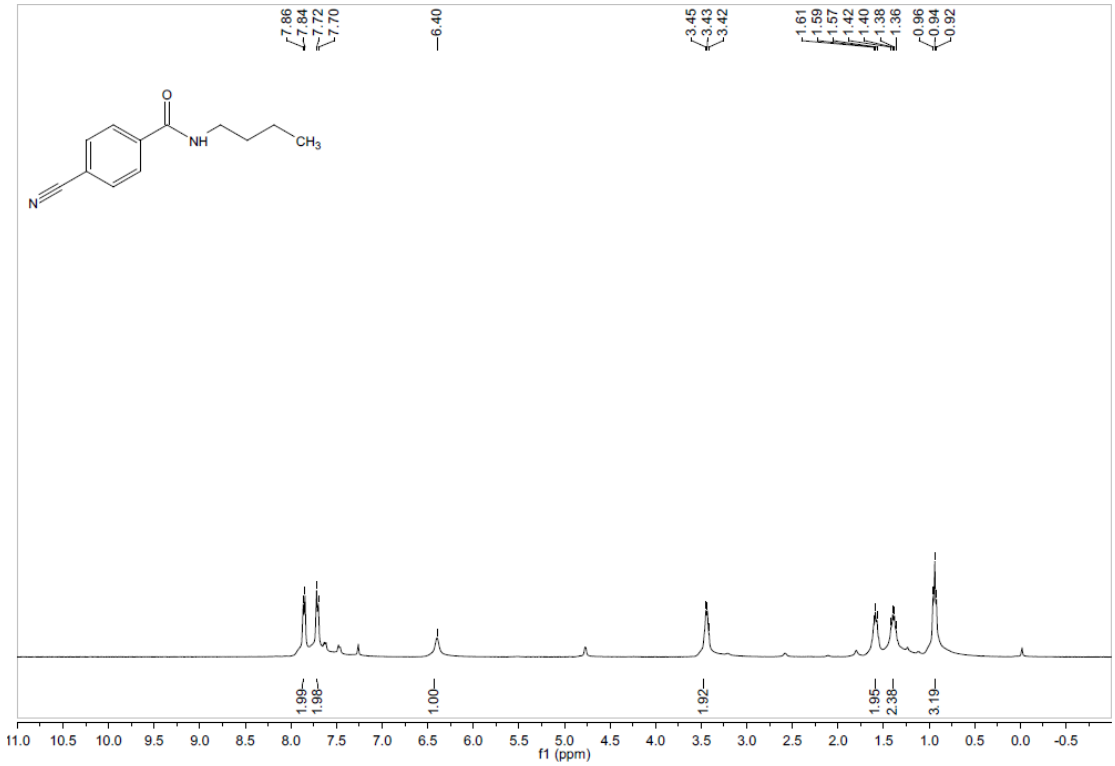
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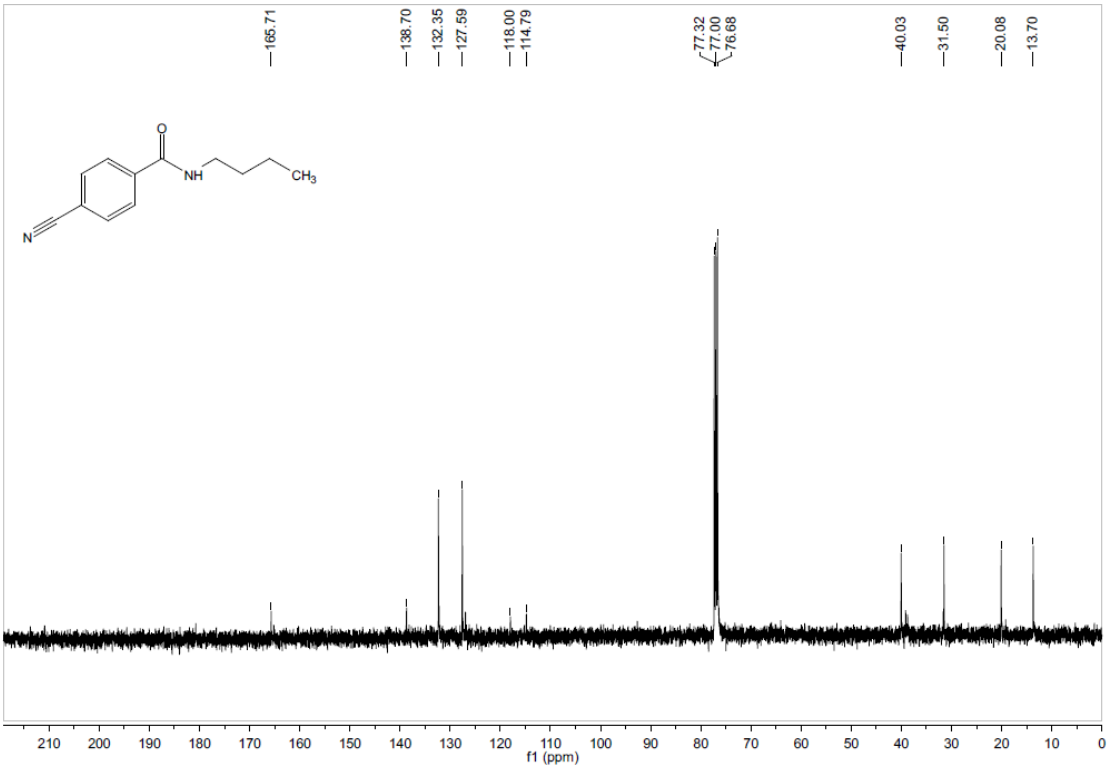
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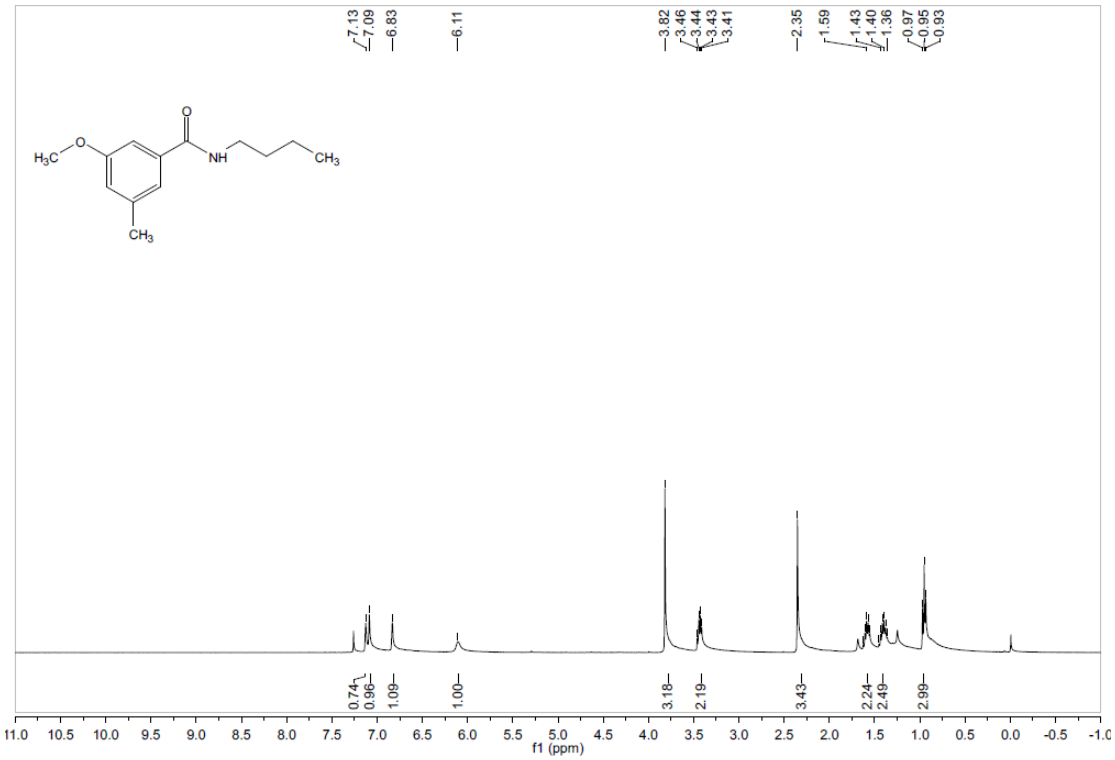
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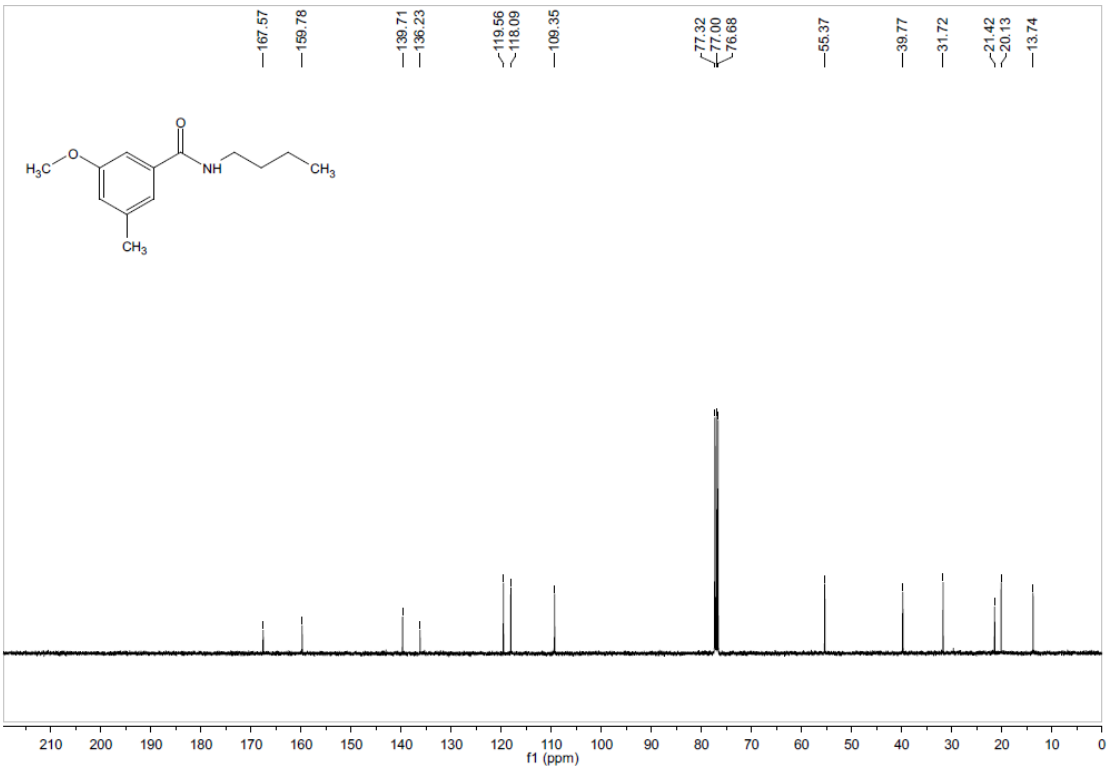
f1 (ppm)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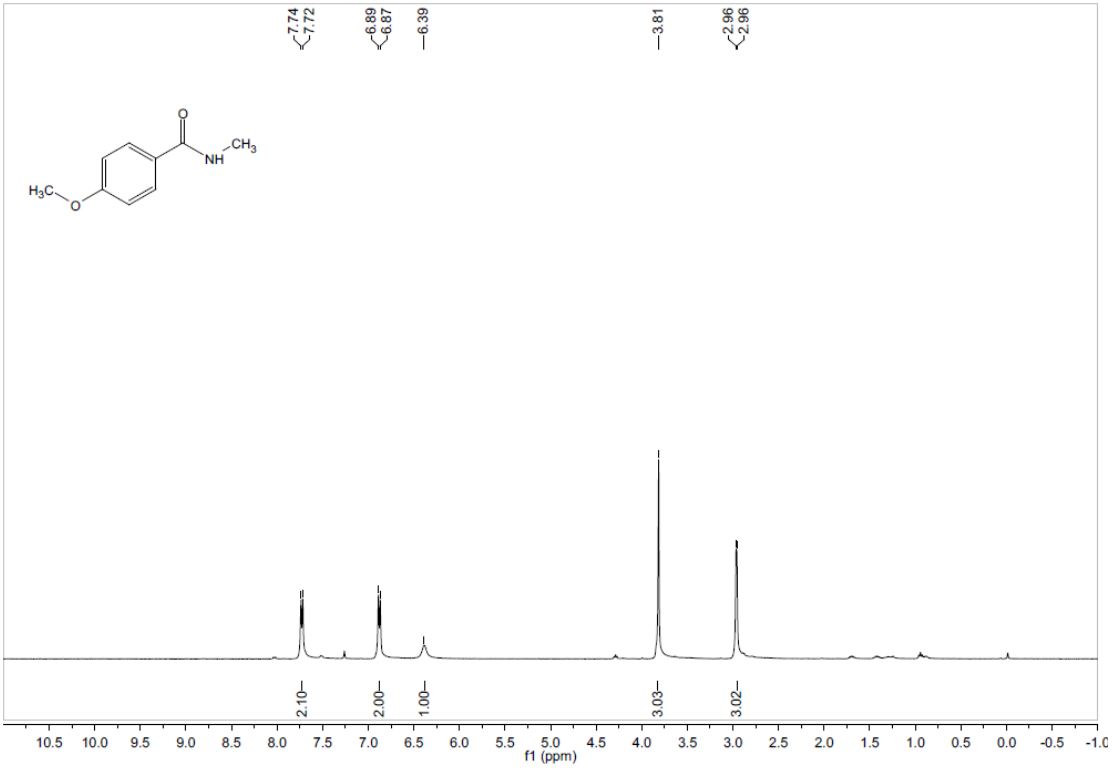
139.71
136.23
119.56
118.09
109.35
77.32
77.00
76.68
55.37
39.77
31.72
21.42
20.13
13.74
O
H3C
O
NH
CH3
CH3
f1 (ppm)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7.72
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6.87
6.39
3.81
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2.96
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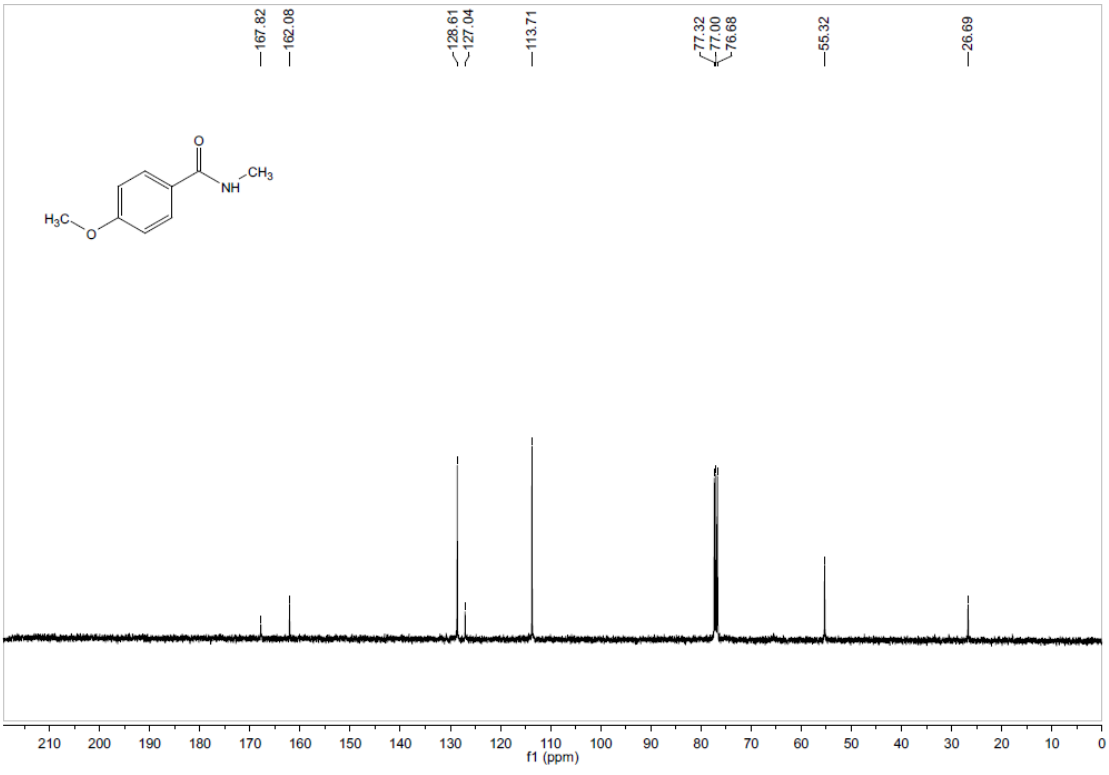
NH
CH3
H3C
O
2.10
2.00
1.00
3.03
3.02
f1 (ppm)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26.69
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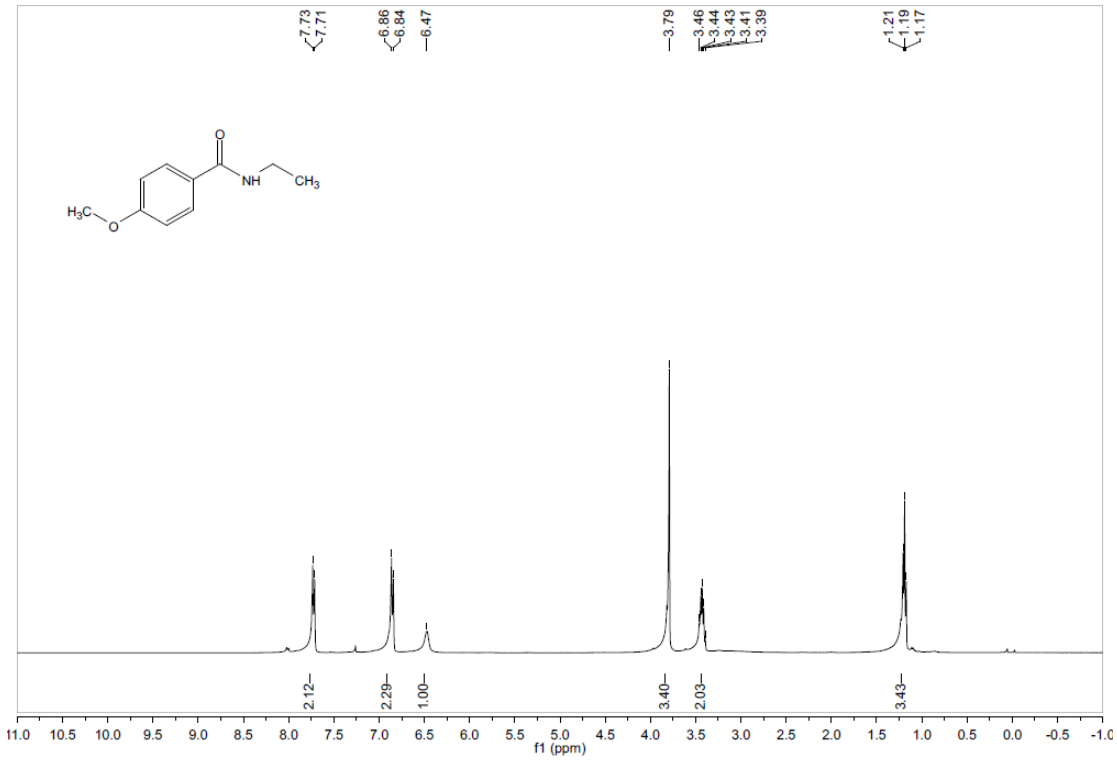
NH
CH3
H3C
O
f1 (ppm)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6.47
3.79
3.46
3.44
3.43
3.41
3.39
1.21
1.19
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CH3
H3C
O
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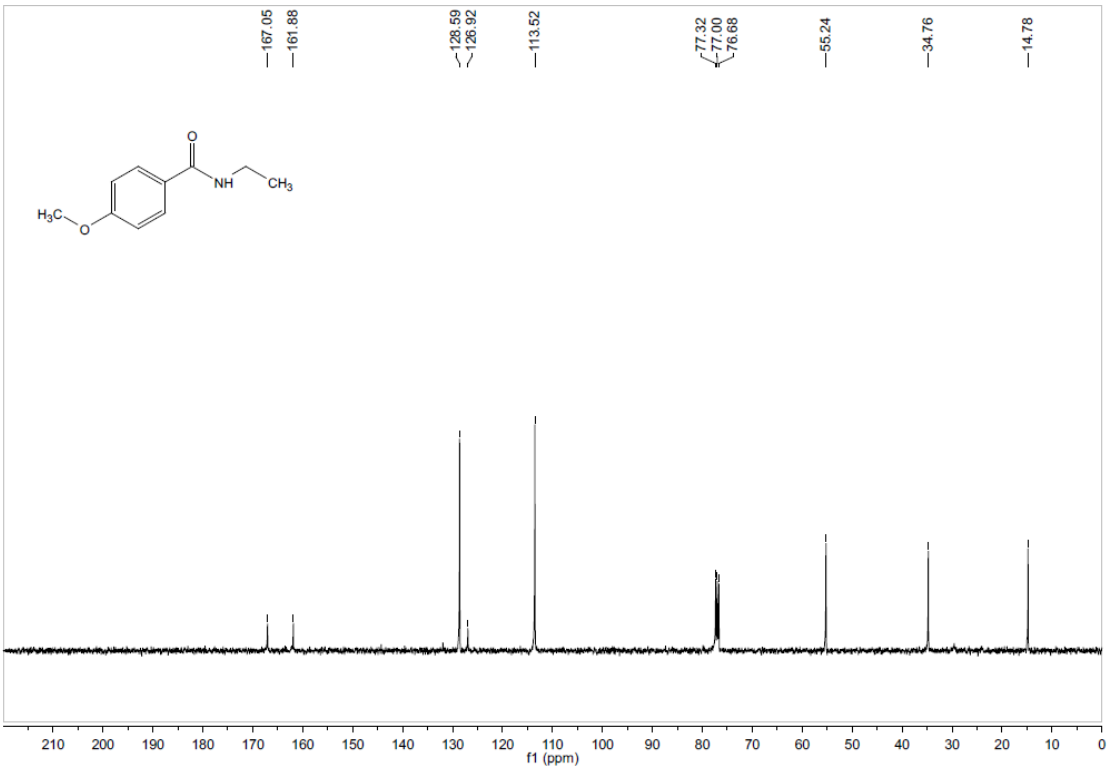
2.29
1.00
3.40
2.03
3.43
f1 (ppm)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76.68
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34.76
14.78
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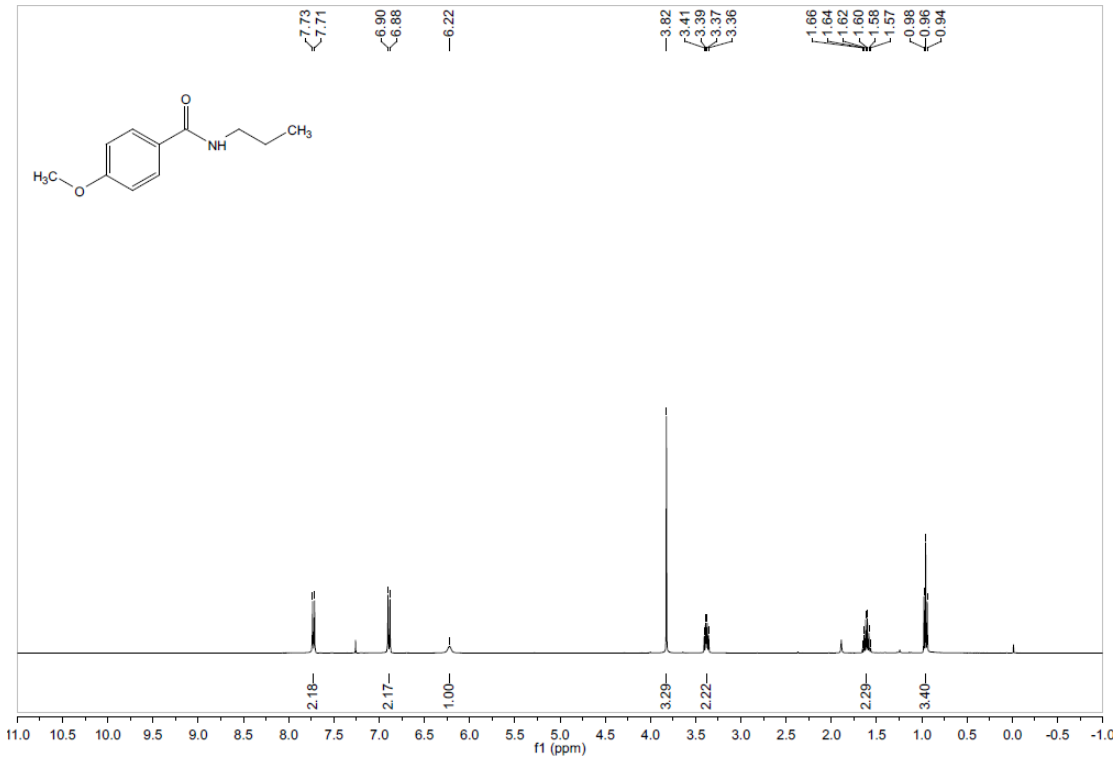
NH
CH3
H3C
O
f1 (ppm)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6.22
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3.41
3.39
3.37
3.36
1.66
1.64
1.62
1.60
1.58
1.57
0.98
0.96
0.94
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NH
CH3
H3C
O
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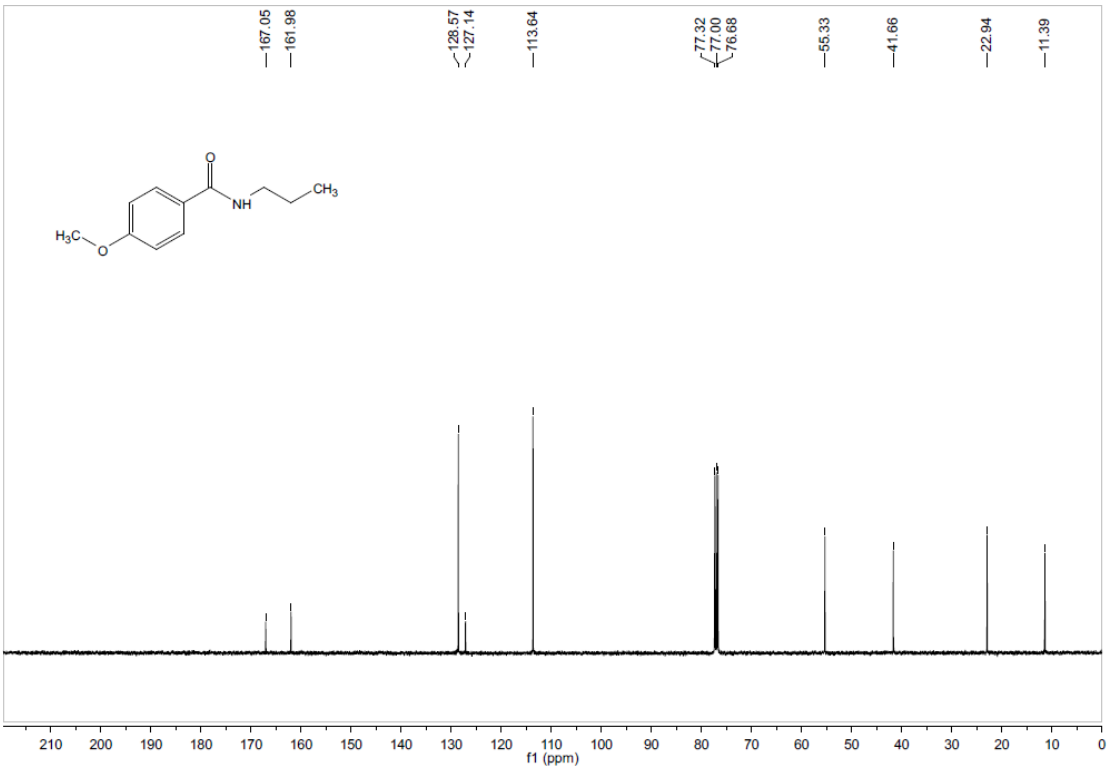
2.17
1.00
3.29
2.22
2.29
3.40
f1 (ppm)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76.68
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22.94
11.39
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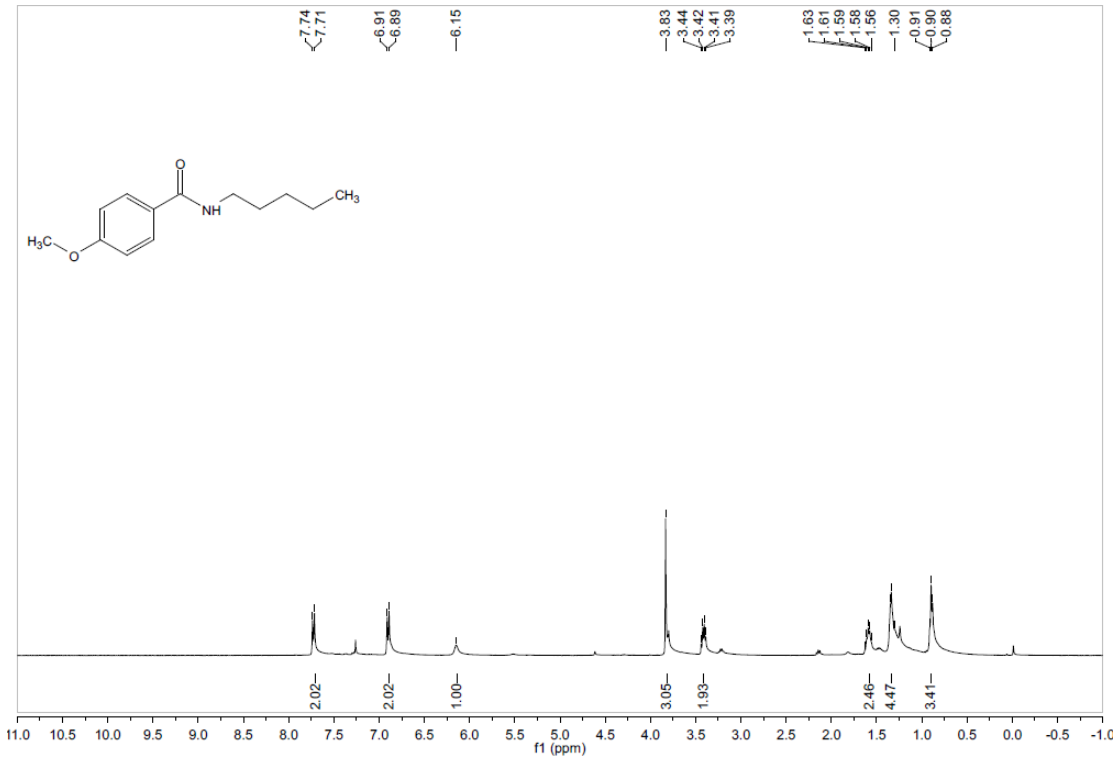
NH
CH3
H3C
O
f1 (ppm)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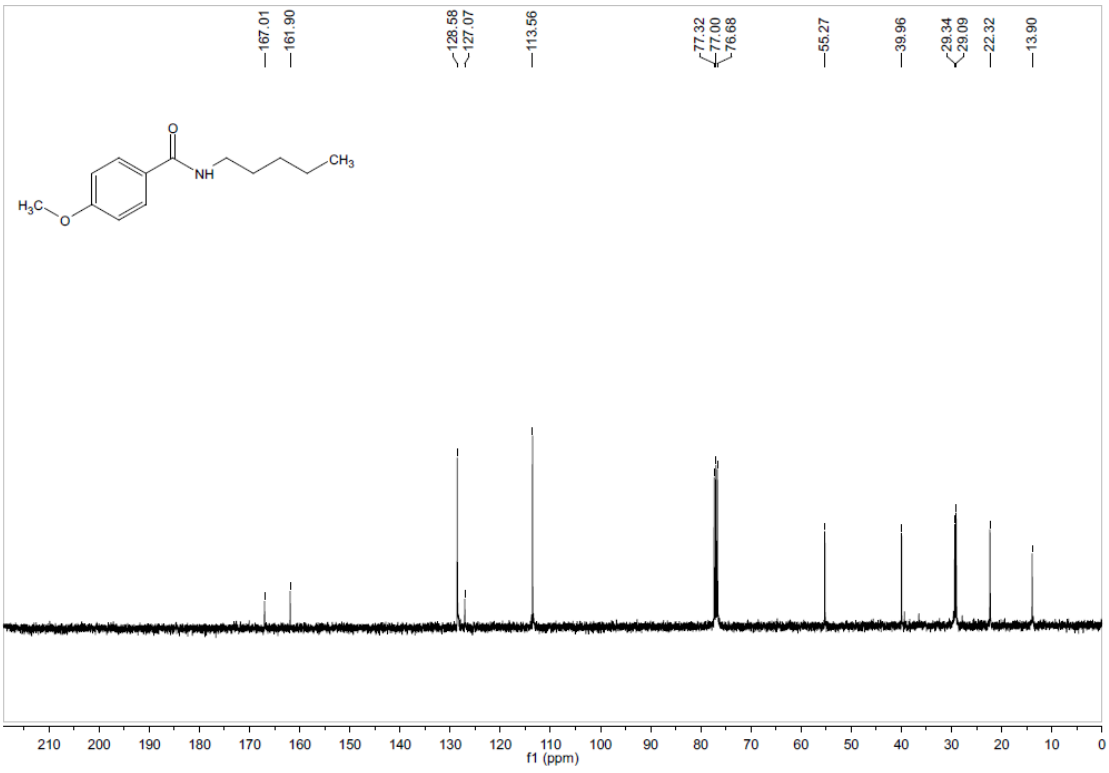
6.89
6.15
3.83
3.44
3.42
3.41
3.39
1.63
1.61
1.59
1.58
1.56
1.30
0.91
0.90
0.88
O
NH
CH3
H3C
O
2.02
2.02
1.00
3.05
1.93
2.46
4.47
3.41
f1 (ppm)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77.00
76.68
55.27
39.96
29.34
29.09
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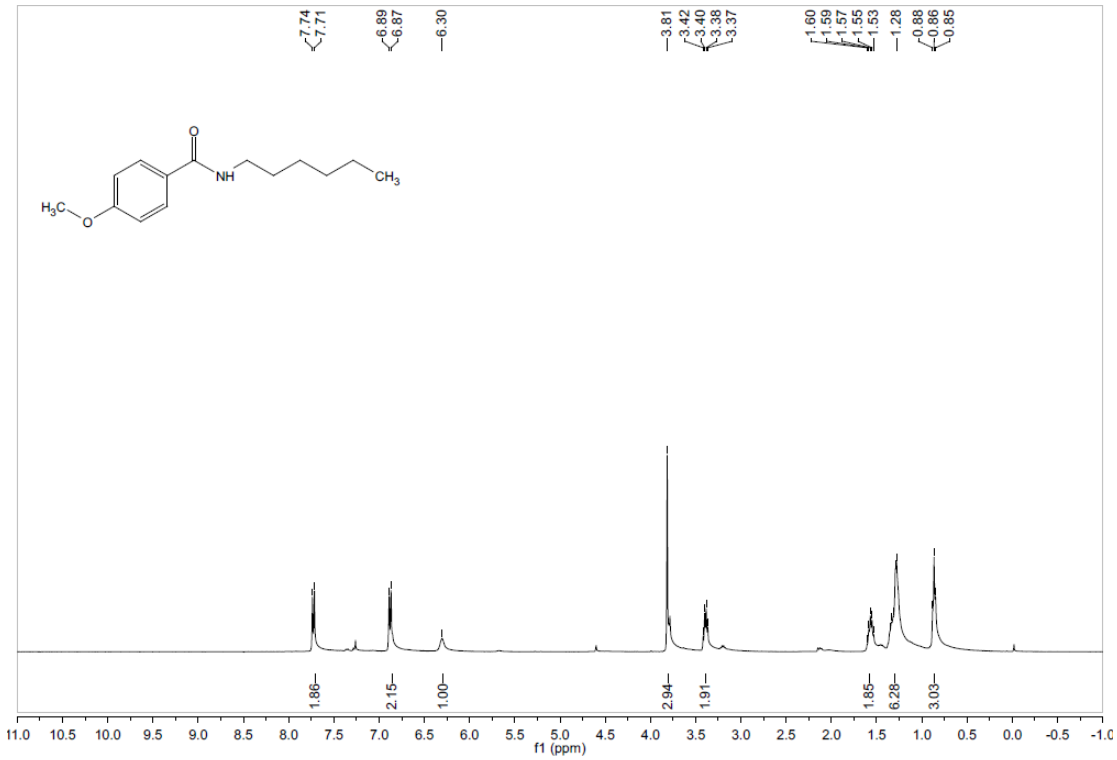
13.90
O
NH
CH3
H3C
O
f1 (ppm)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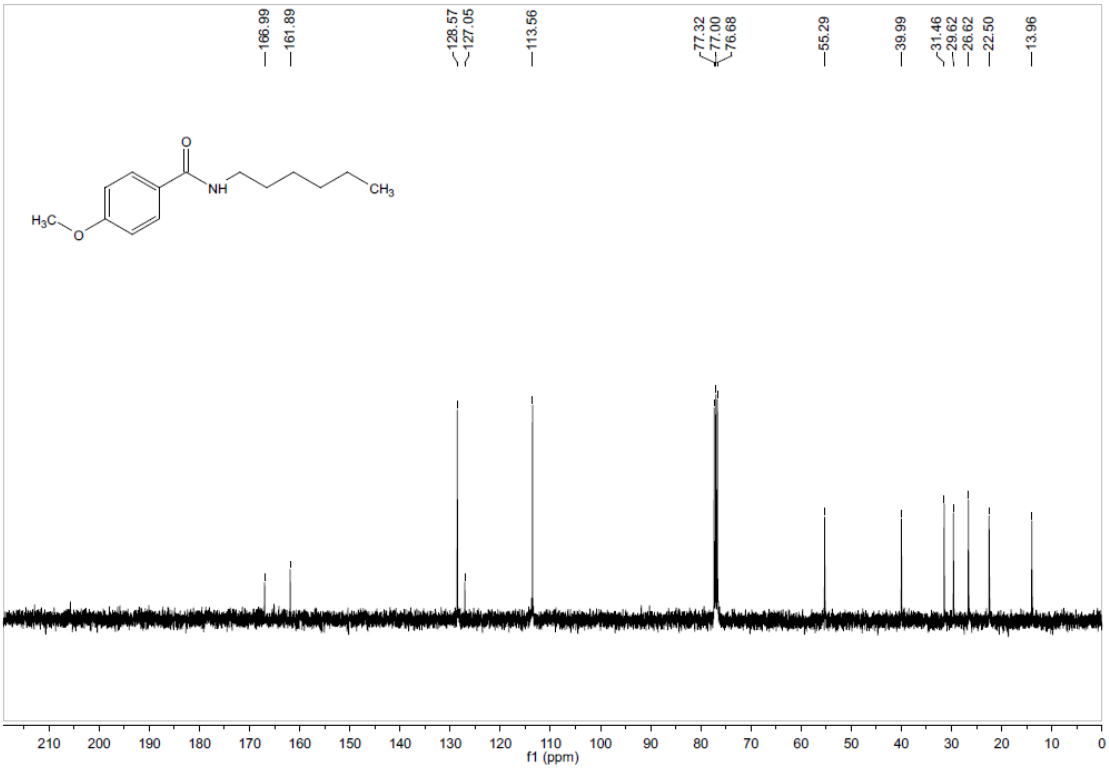
7.71
6.89
6.87
6.30
3.81
3.42
3.40
3.38
3.37
1.60
1.59
1.57
1.55
1.53
1.28
0.88
0.86
0.85
O
NH
CH3
H3C
O
1.86
2.15
1.00
2.94
1.91
1.85
6.28
3.03
f1 (ppm)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161.89
128.57
127.05
113.56
77.32
77.00
76.68
55.29
39.99
31.46
29.62
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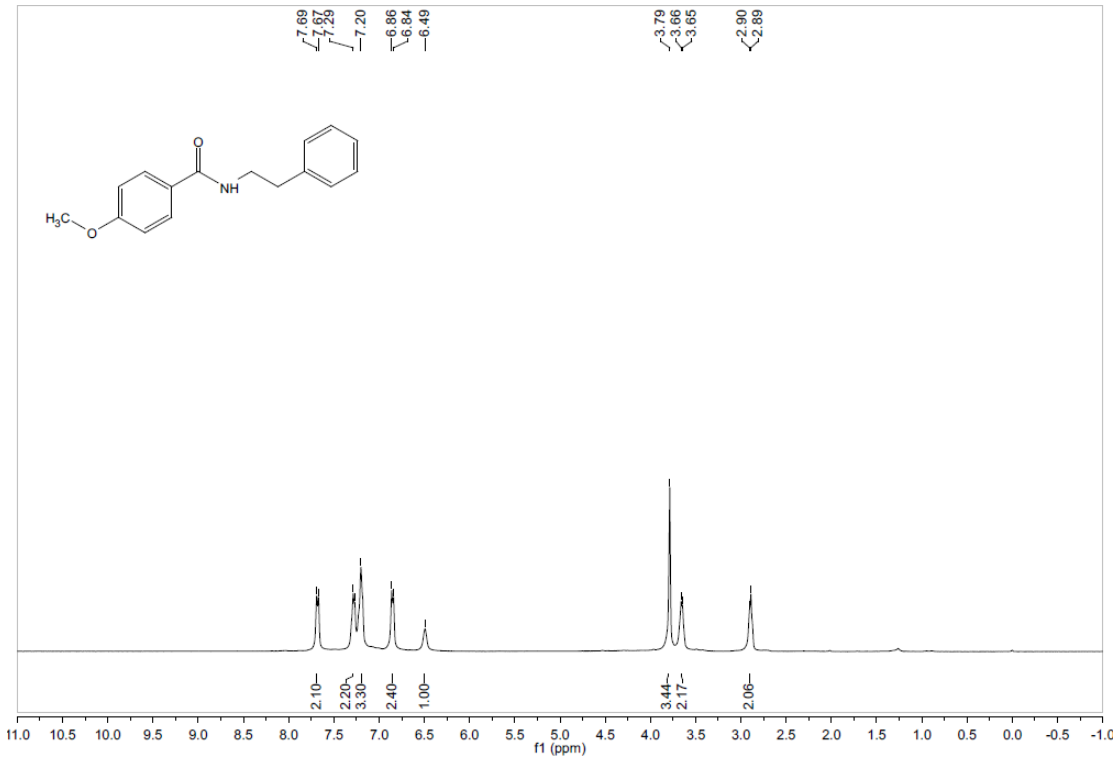
13.96
O
NH
CH3
H3C
O
f1 (ppm)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6.84
6.49
3.79
3.66
3.65
2.90
2.89
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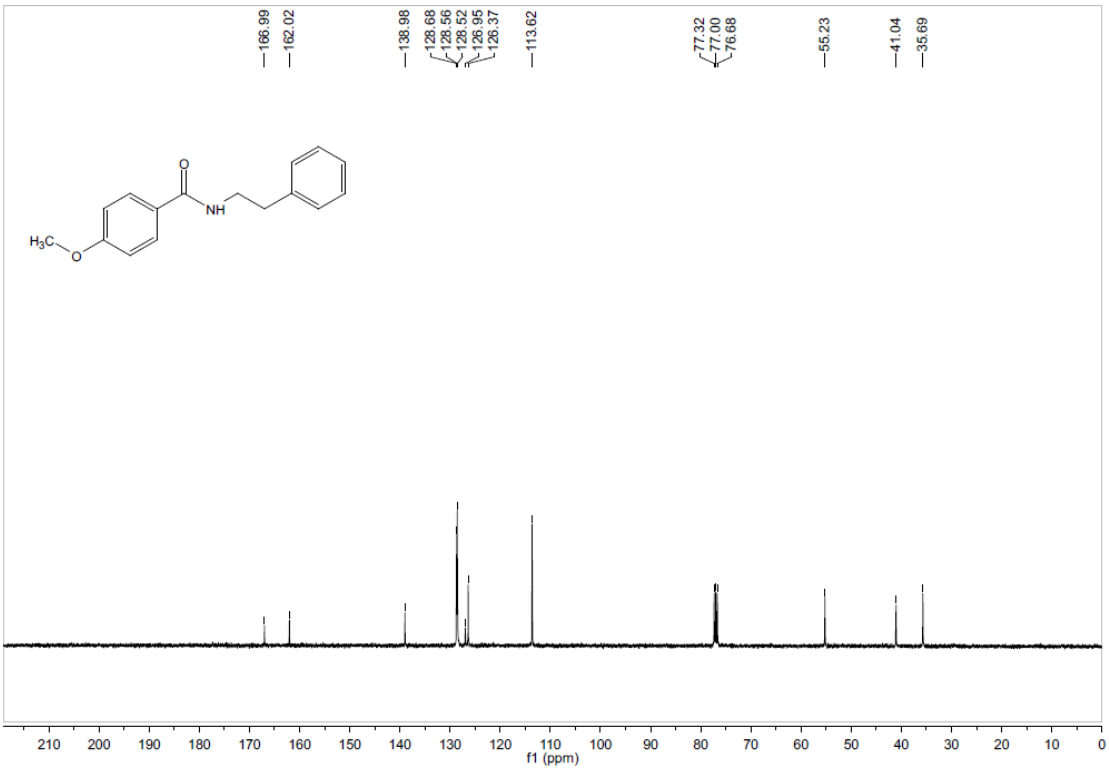
NH
H3C
O
2.10
2.20
3.30
2.40
1.00
3.44
2.17
2.06
f1 (ppm)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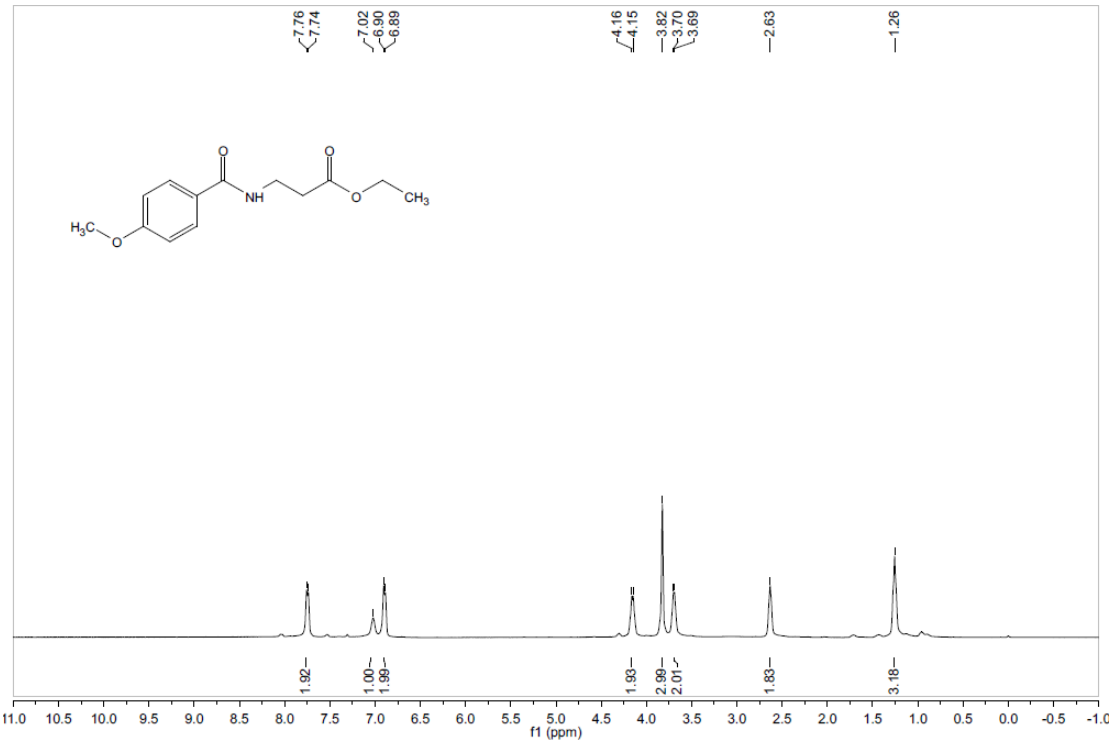
77.00
76.68
55.23
41.04
35.69
O
NH
H3C
O
f1 (ppm)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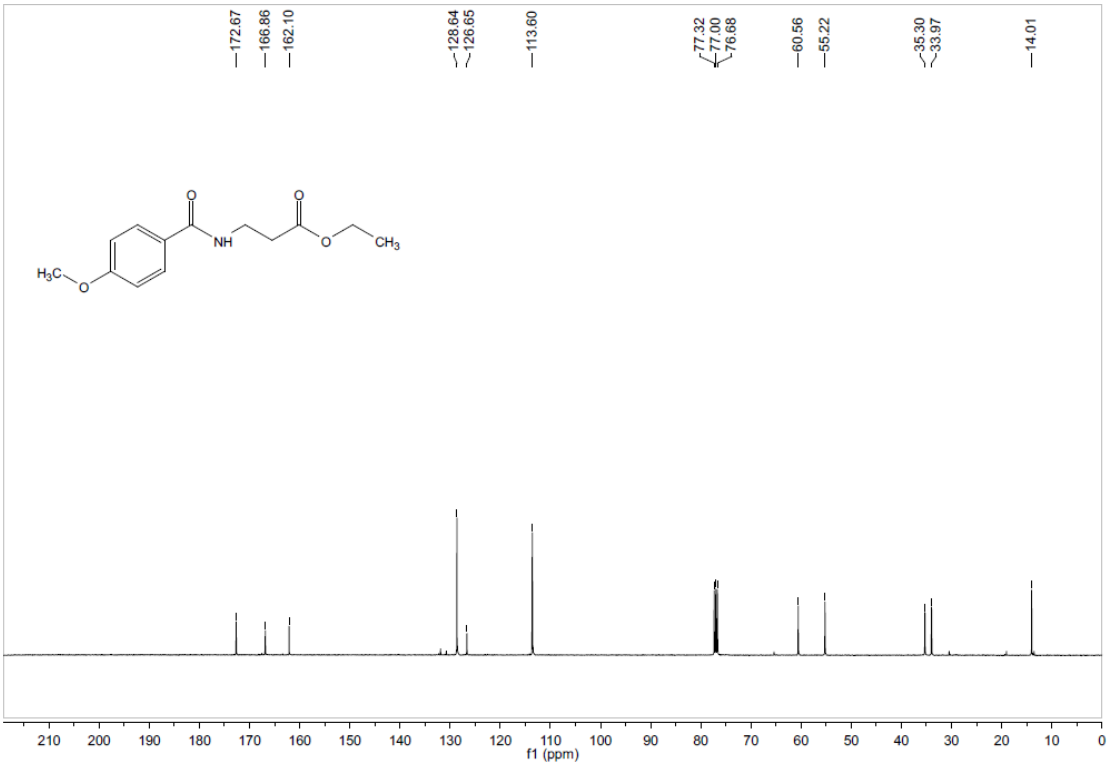
6.89
4.16
4.15
3.82
3.70
3.69
2.63
1.26
O
NH
O
O
CH3
H3C
O
1.92
1.00
1.99
1.93
2.99
2.01
1.83
3.18
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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77.00
76.68
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55.22
35.30
33.97
14.01
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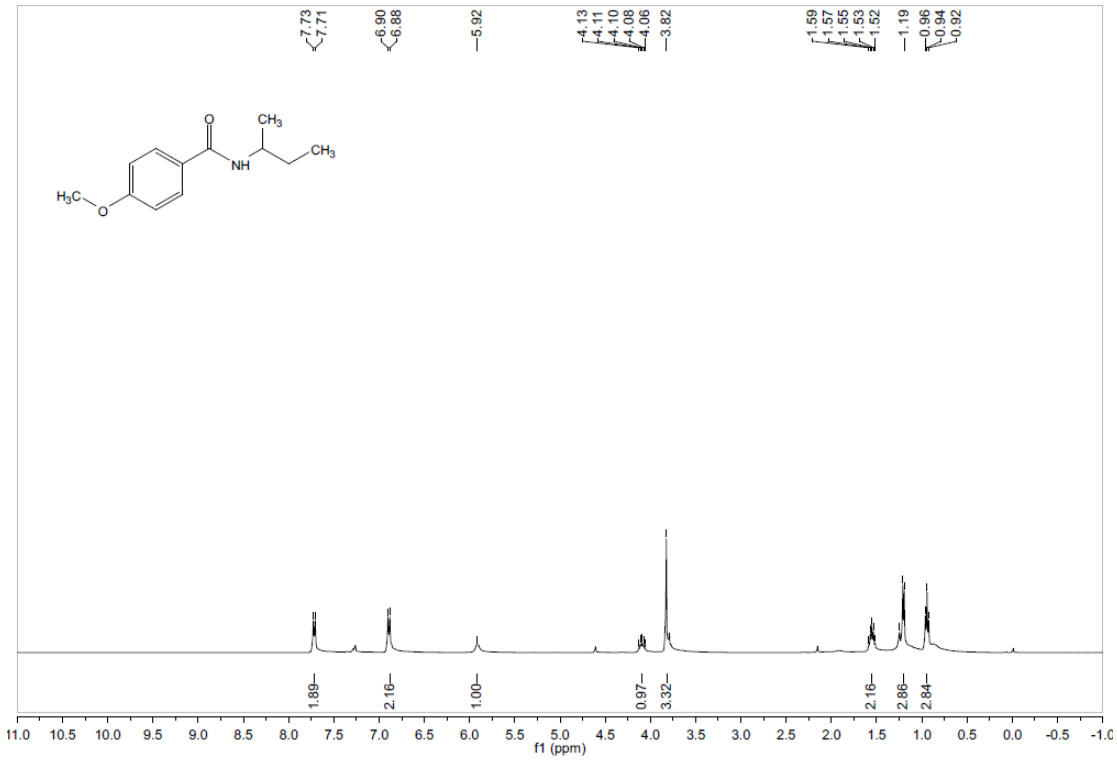
O
O
CH3
H3C
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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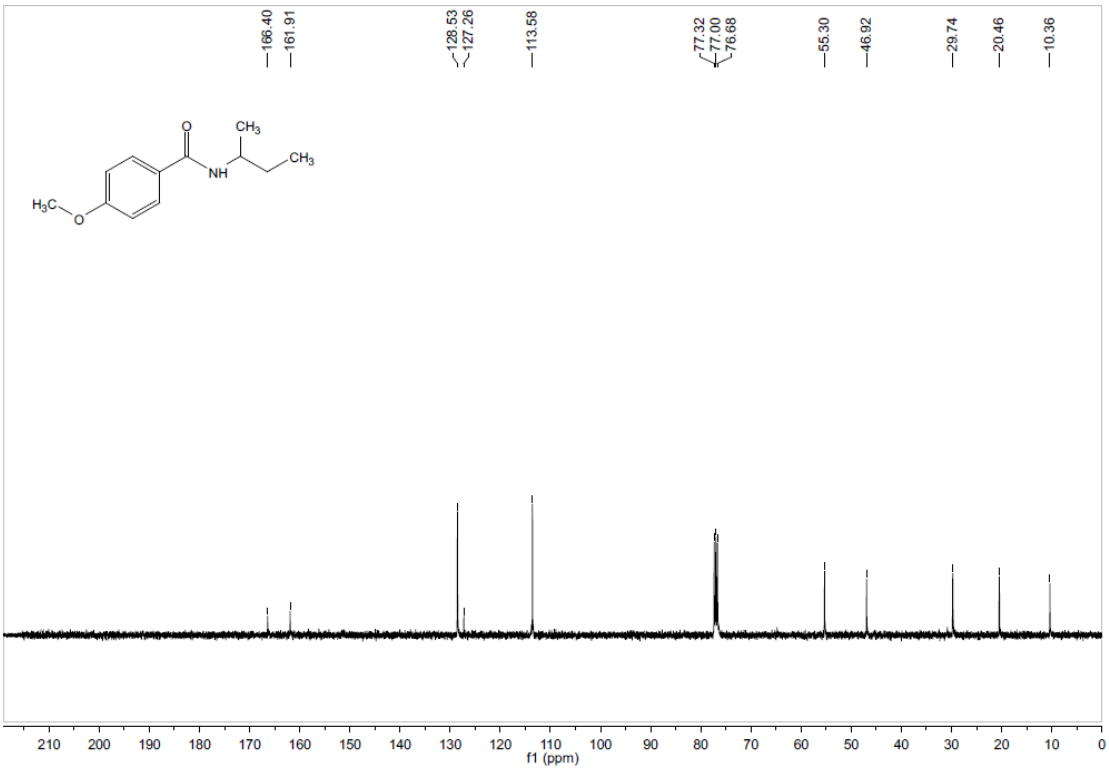
5.92
4.13
4.11
4.10
4.08
4.06
3.82
1.59
1.57
1.55
1.53
1.52
1.19
0.96
0.94
0.92
O
CH3
NH
CH3
H3C
O
1.89
2.16
1.00
0.97
3.32
2.16
2.86
2.84
f1 (ppm)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77.00
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46.92
29.74
20.46
10.36
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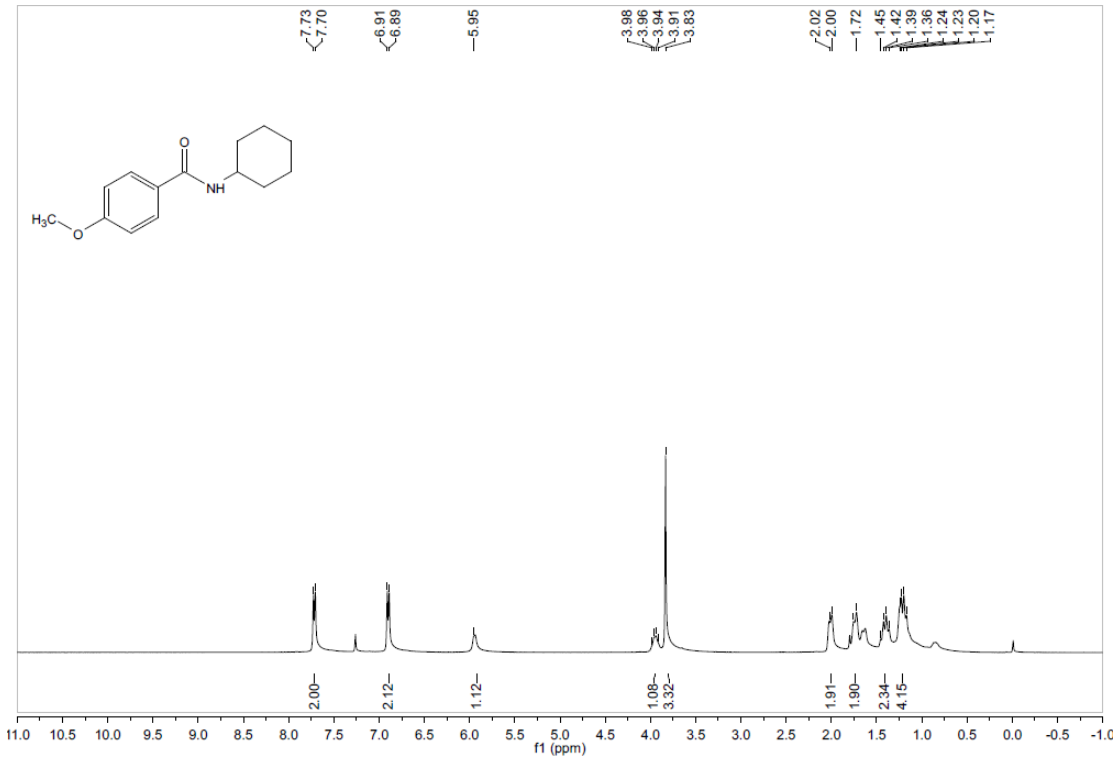
CH3
NH
CH3
H3C
O
f1 (ppm)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3.94
3.91
3.83
2.02
2.00
1.72
1.45
1.42
1.39
1.36
1.24
1.23
1.20
1.17
O
NH
H3C
O
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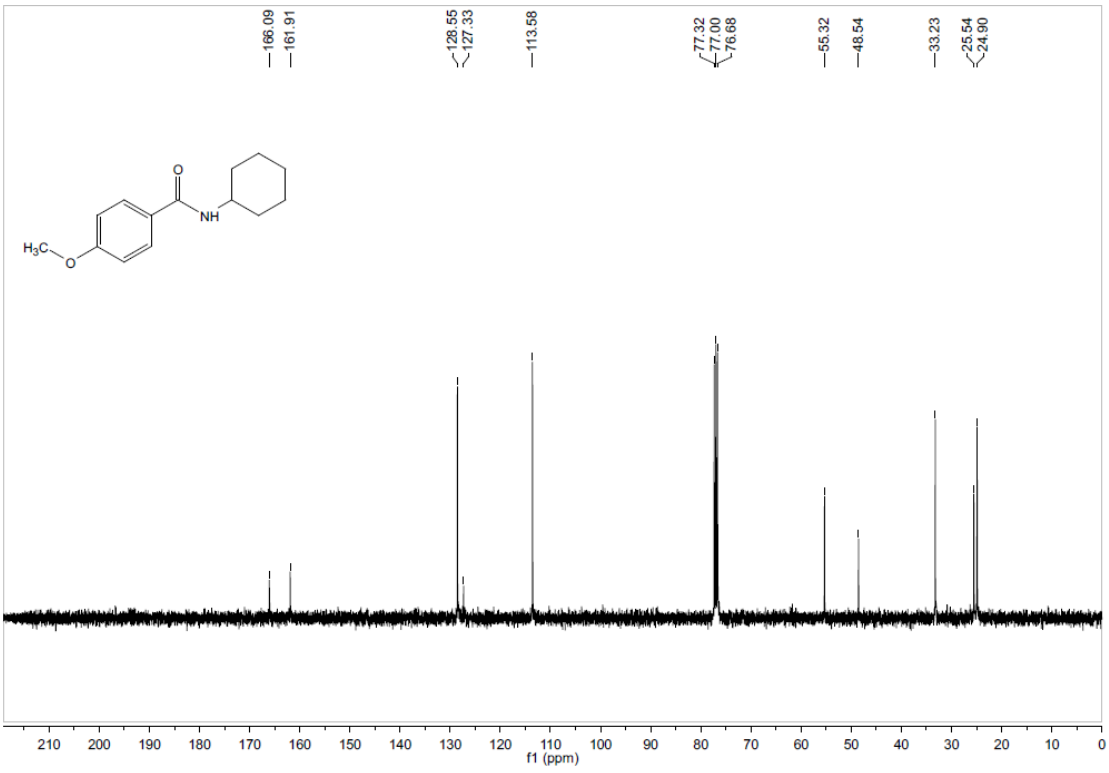
2.12
1.12
1.08
3.32
1.91
1.90
2.34
4.15
f1 (ppm)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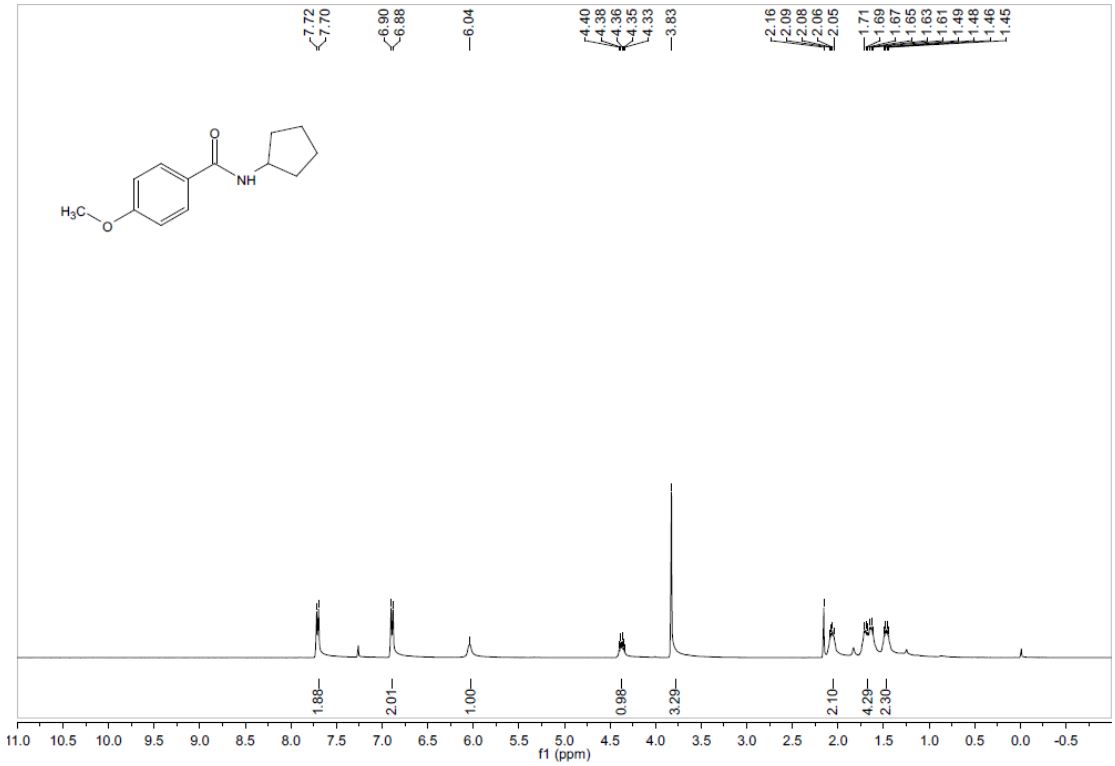
NH
H3C
O
f1 (ppm)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4.35
4.33
3.83
2.16
2.09
2.08
2.06
2.05
1.71
1.69
1.67
1.65
1.63
1.61
1.49
1.48
1.46
1.45
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O
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1.00
0.98
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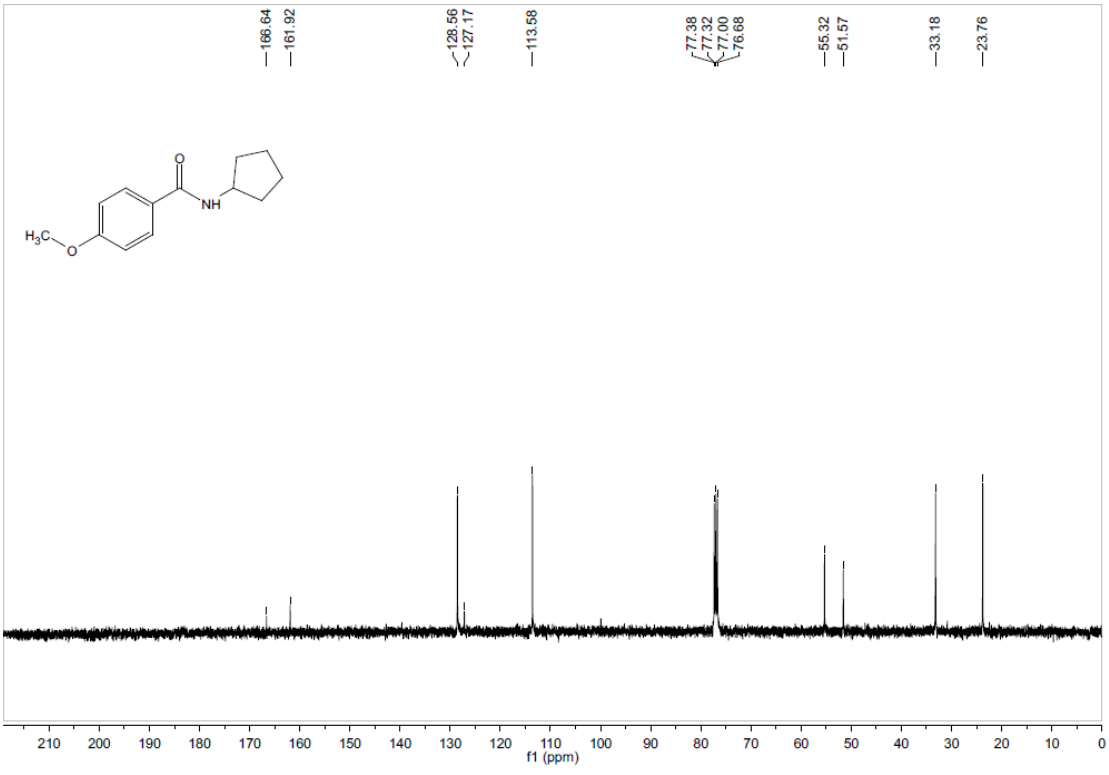
2.10
4.29
2.30
f1 (ppm)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33.18
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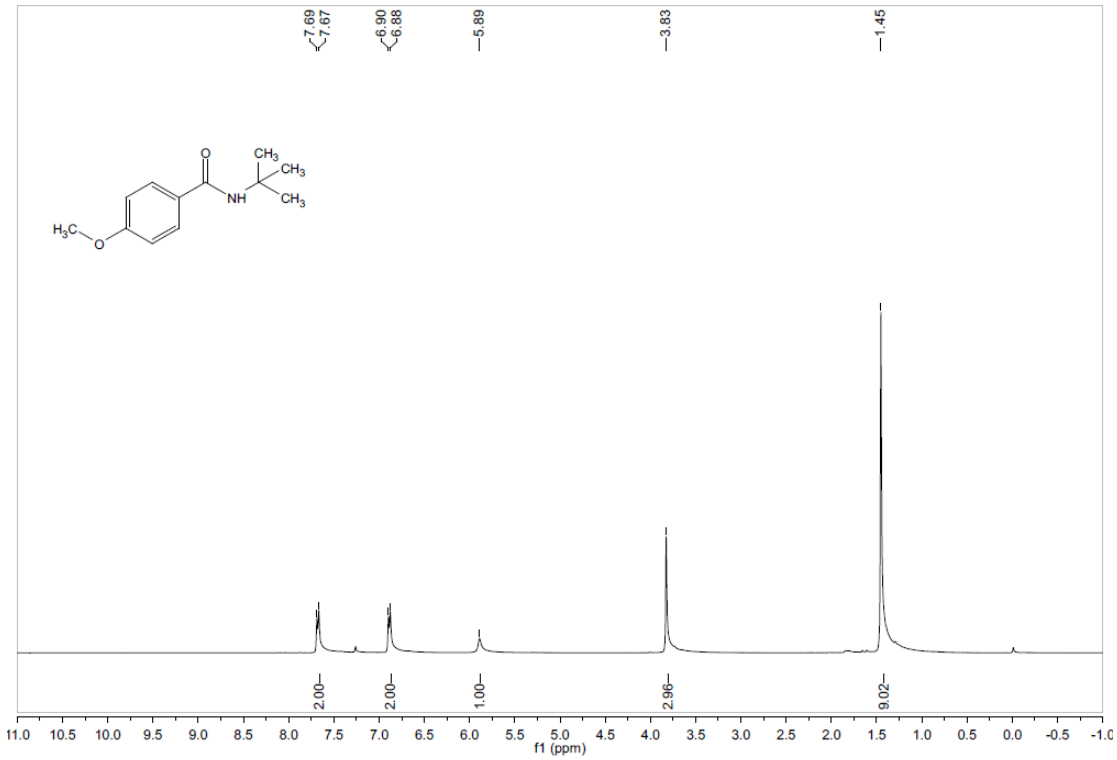
O
NH
H3C
O
f1 (ppm)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6.88
5.89
3.83
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CH3
CH3
NH
CH3
H3C
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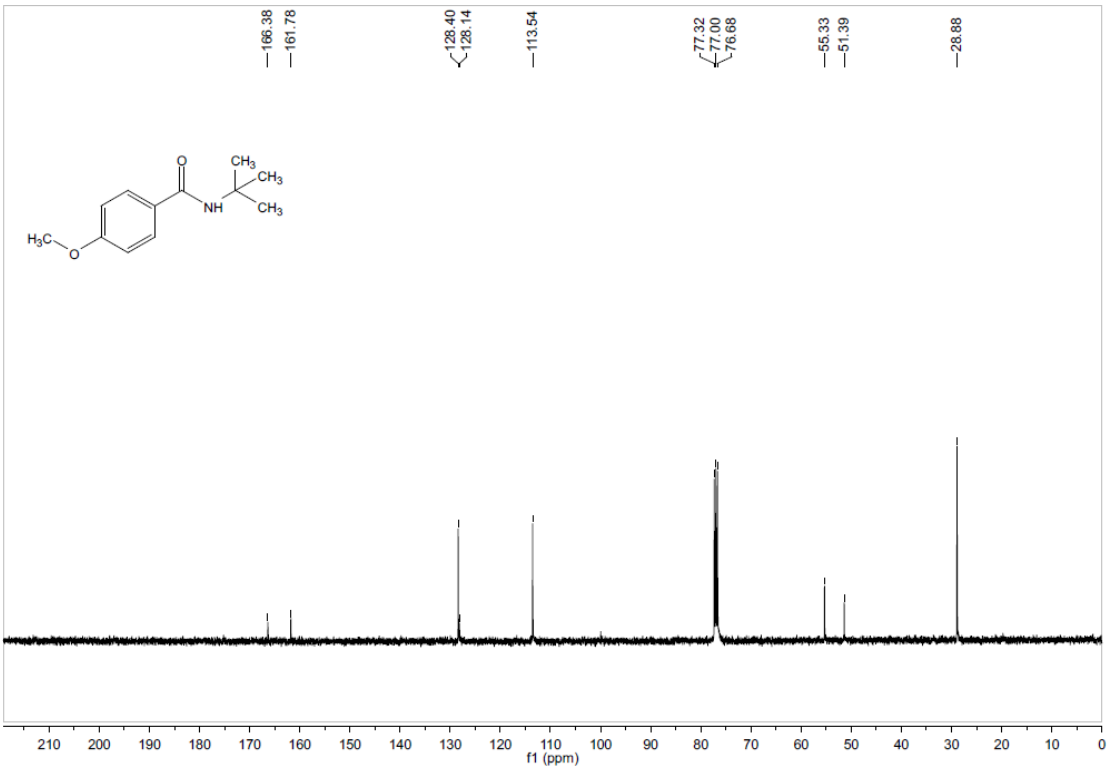
2.00
1.00
2.96
9.02
f1 (ppm)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51.39
28.88
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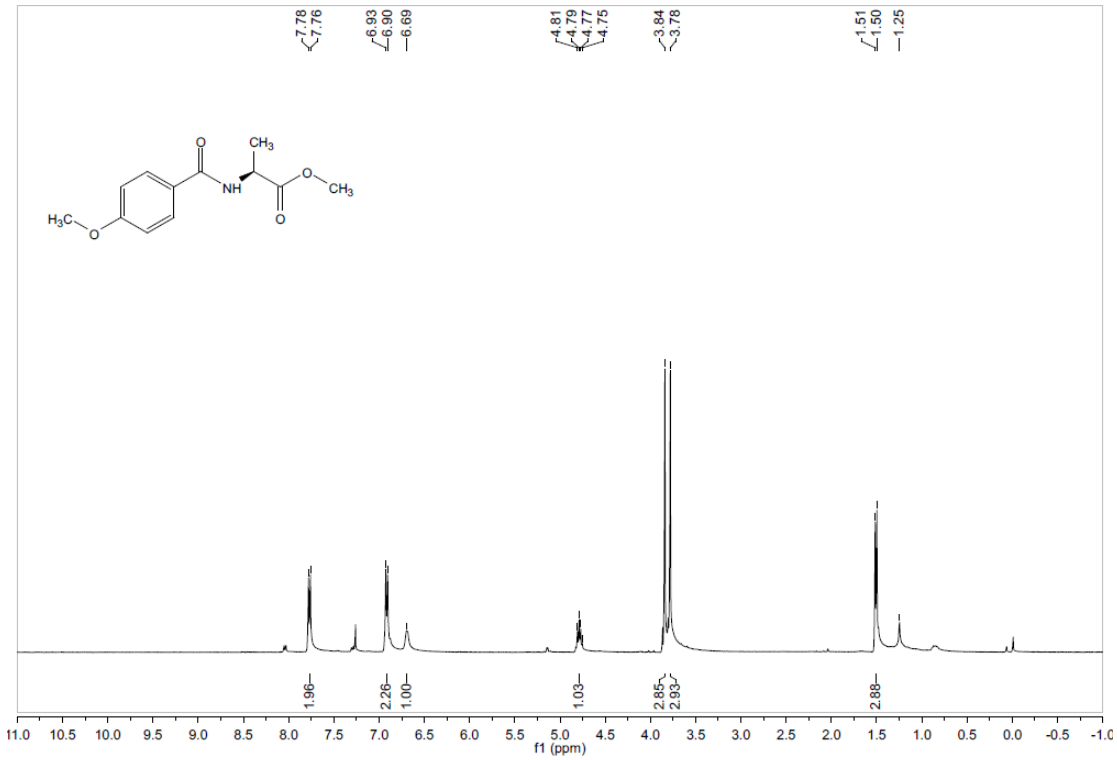
CH3
NH
CH3
H3C
O
f1 (ppm)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4.77
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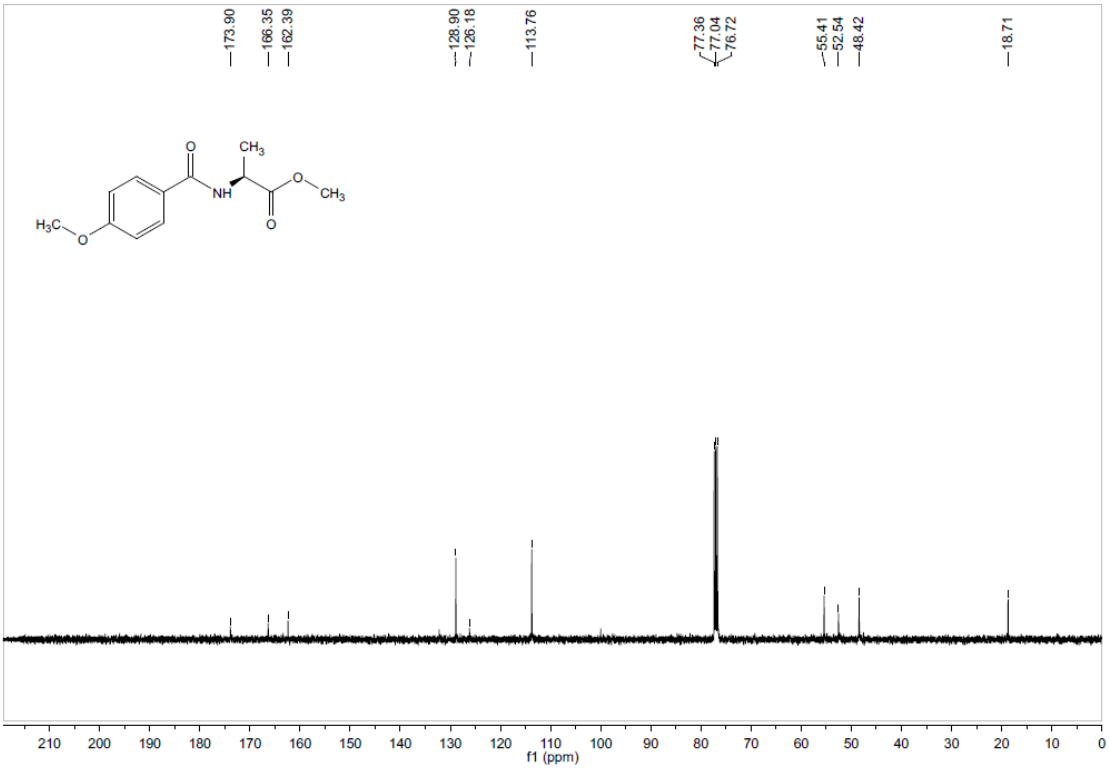
3.84
3.78
1.51
1.50
1.25
O
CH3
NH
O
O
CH3
H3C
O
1.96
2.26
1.00
1.03
2.85
2.93
2.88
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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76.72
55.41
52.54
48.42
18.71
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CH3
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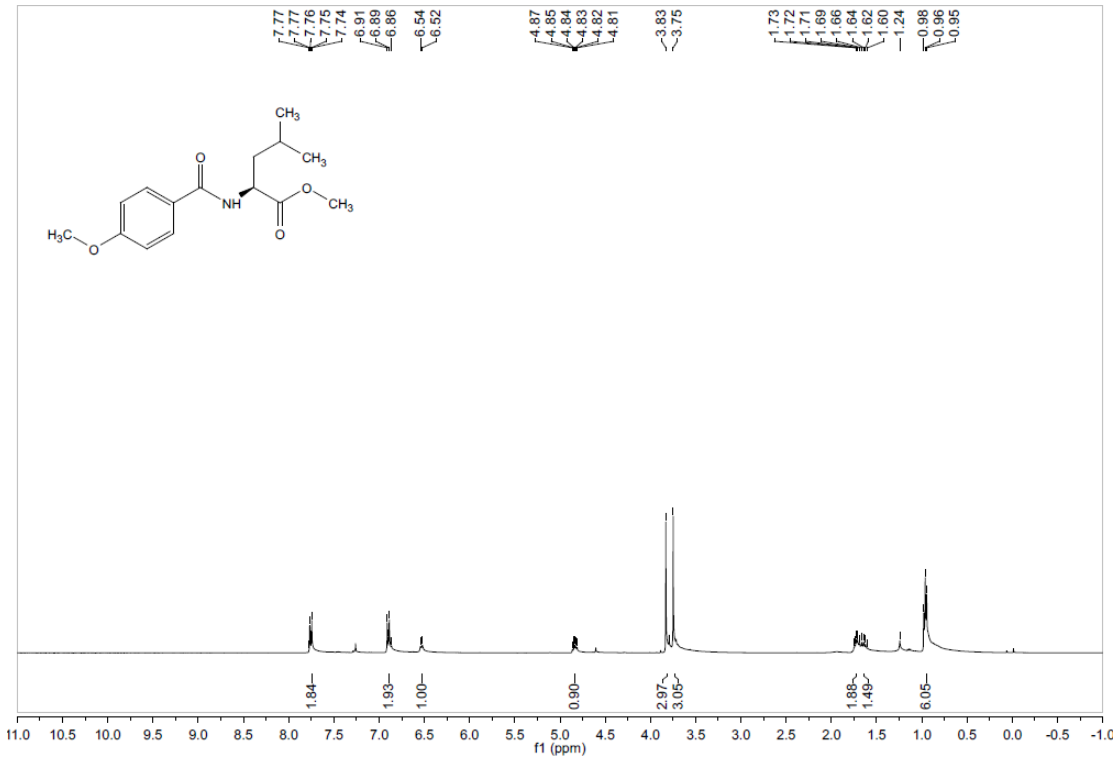
O
O
CH3
H3C
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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4.83
4.82
4.81
3.83
3.75
1.73
1.72
1.71
1.69
1.66
1.64
1.62
1.60
1.24
0.98
0.96
0.95
CH3
CH3
O
NH
O
CH3
H3C
O
O
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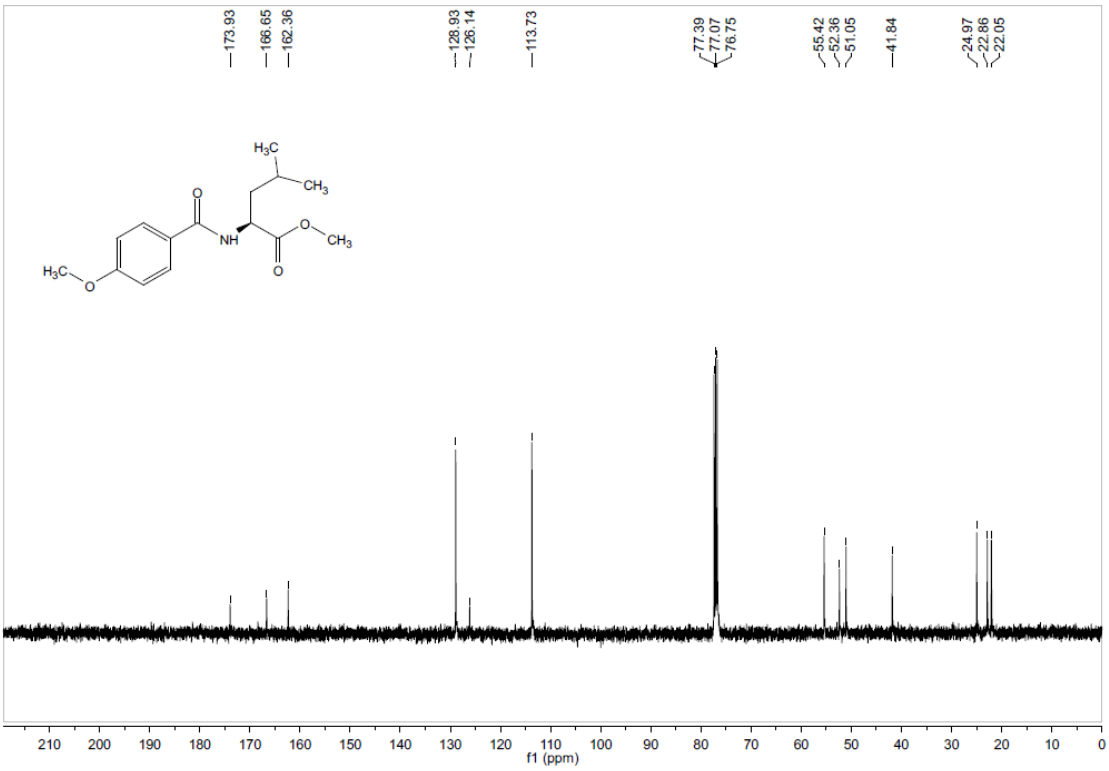
1.93
1.00
0.90
2.97
3.05
1.88
1.49
6.05
f1 (ppm)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55.42
52.36
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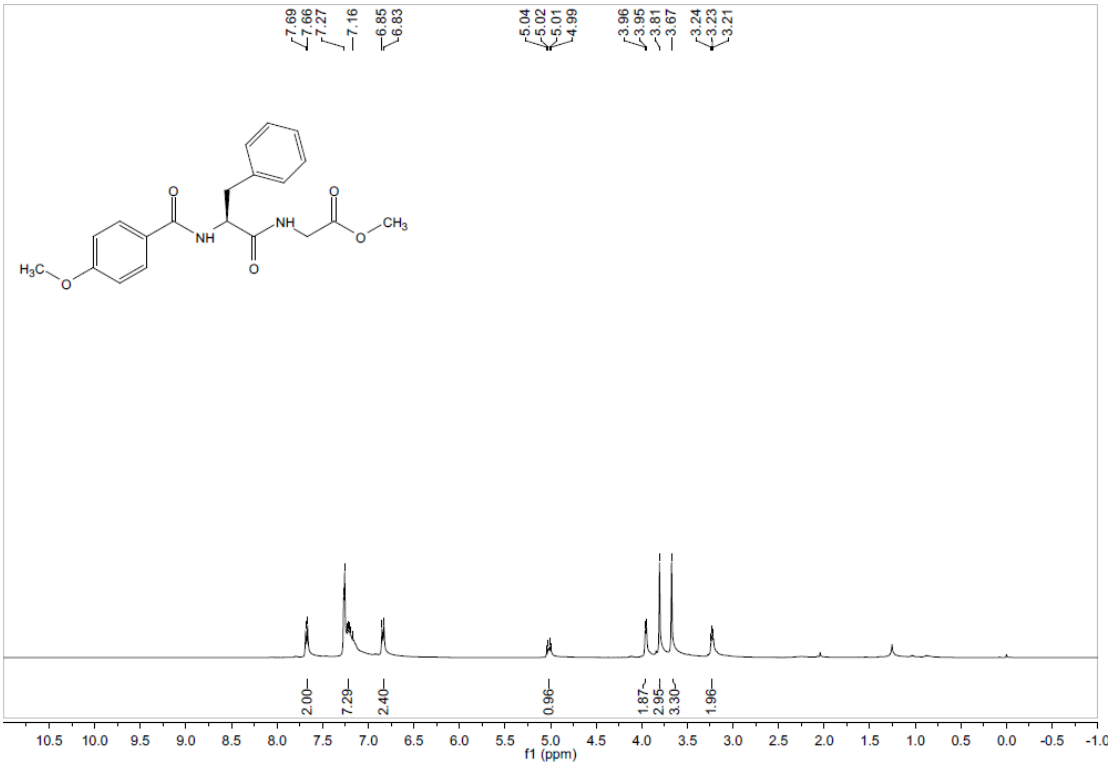
24.97
22.86
22.05
H3C
CH3
O
NH
O
CH3
H3C
O
f1 (ppm)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3.67
3.24
3.23
3.21
NH
NH
O
O
O
O
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H3C
2.00
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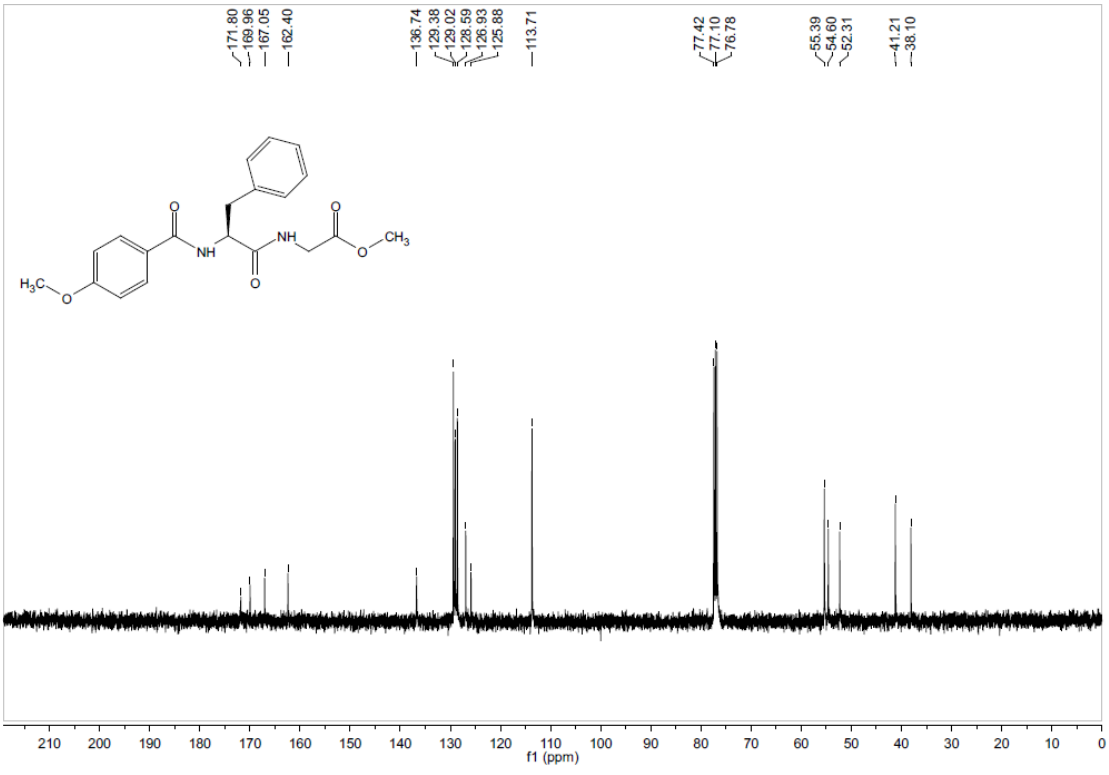
0.96
1.87
2.95
3.30
1.96
f1 (ppm)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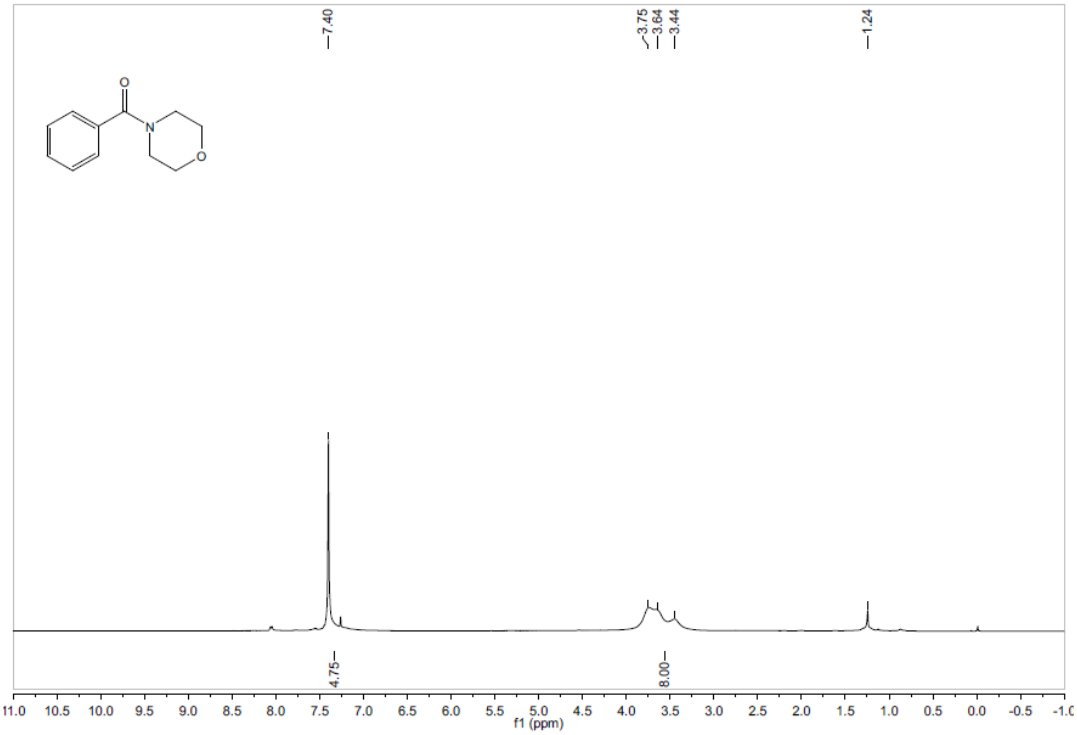
125.88
113.71
77.42
77.10
76.78
55.39
54.60
52.31
41.21
38.10
NH
NH
O
O
O
O
CH3
H3C
f1 (ppm)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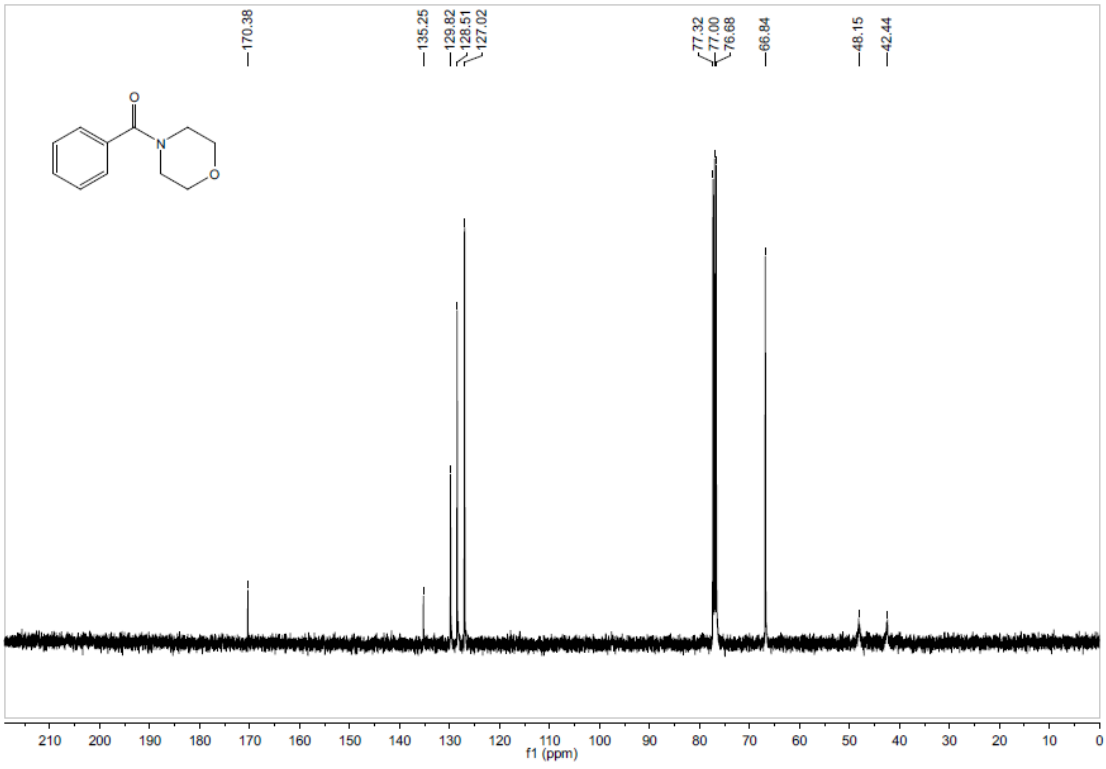
8.00
f1 (ppm)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42.44
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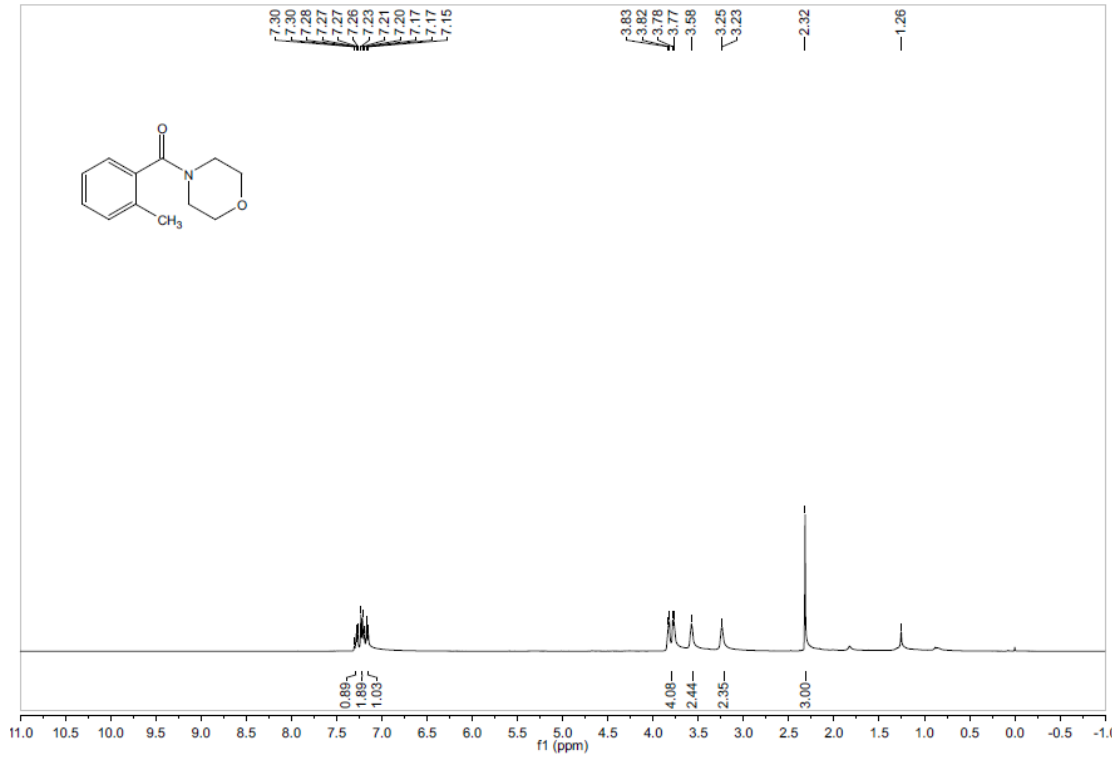
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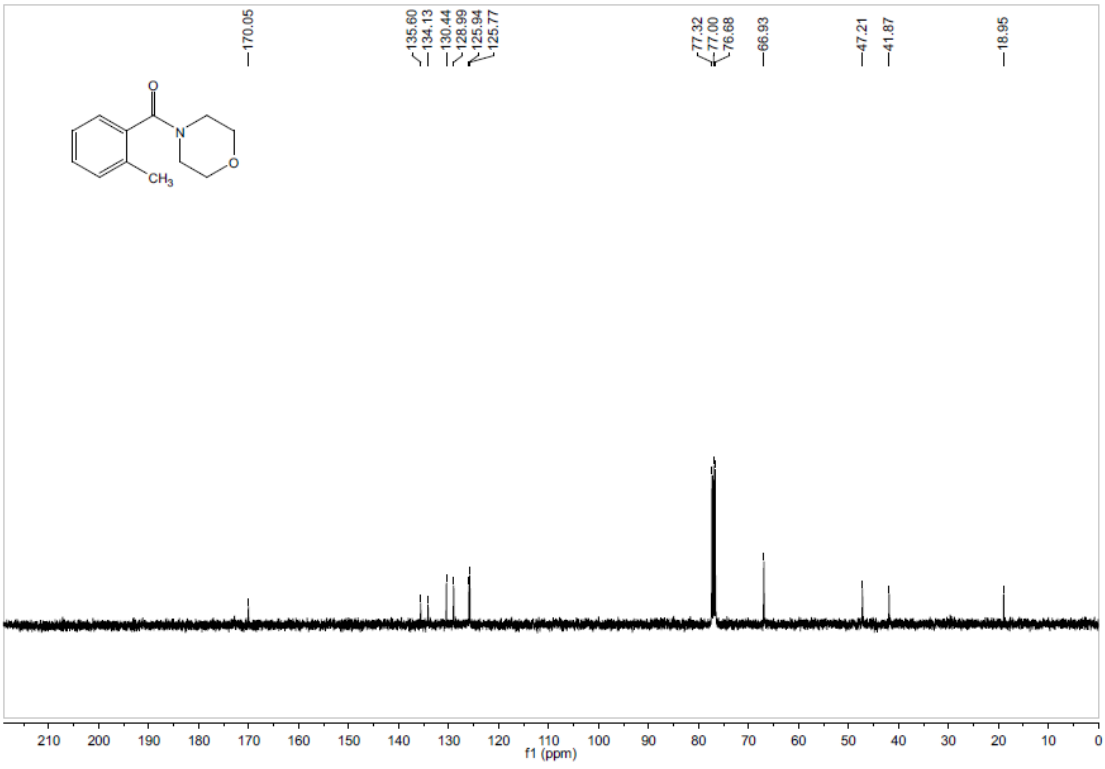
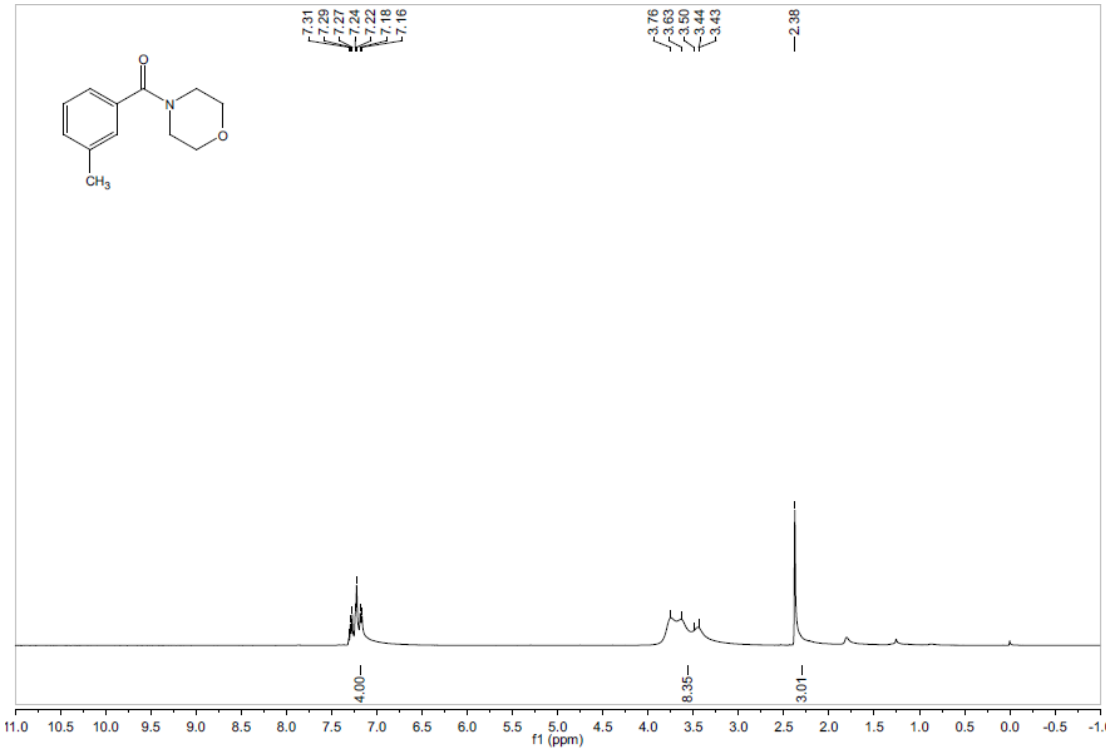
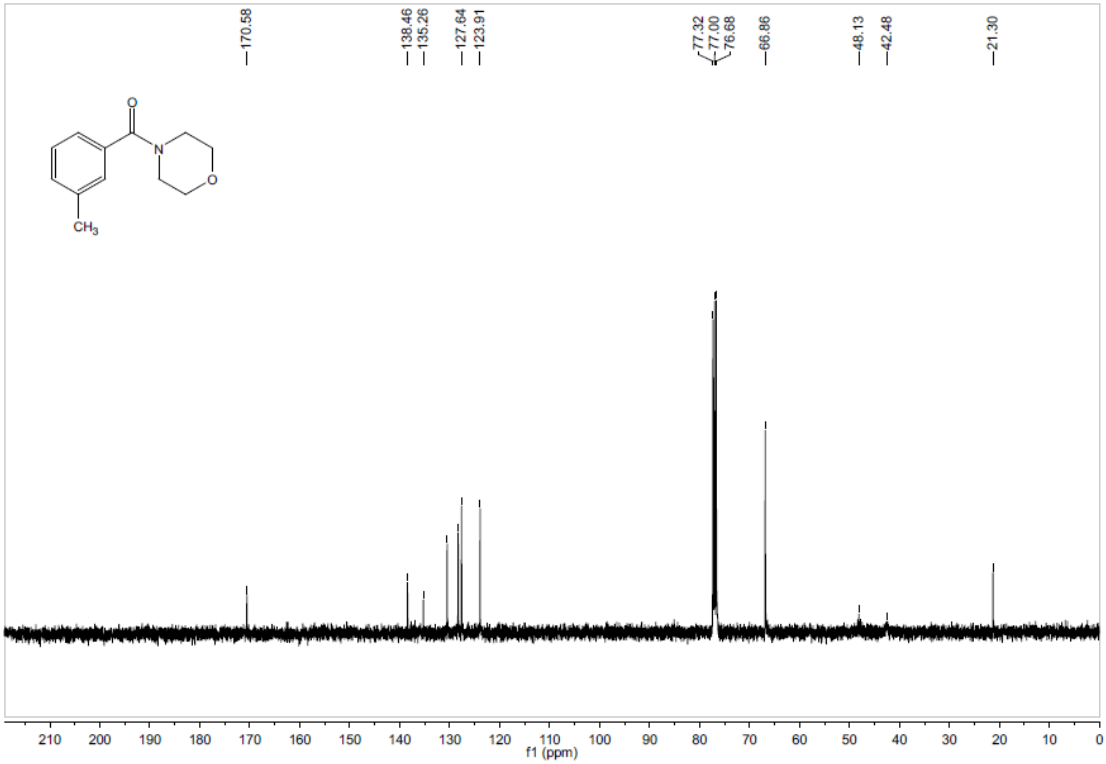
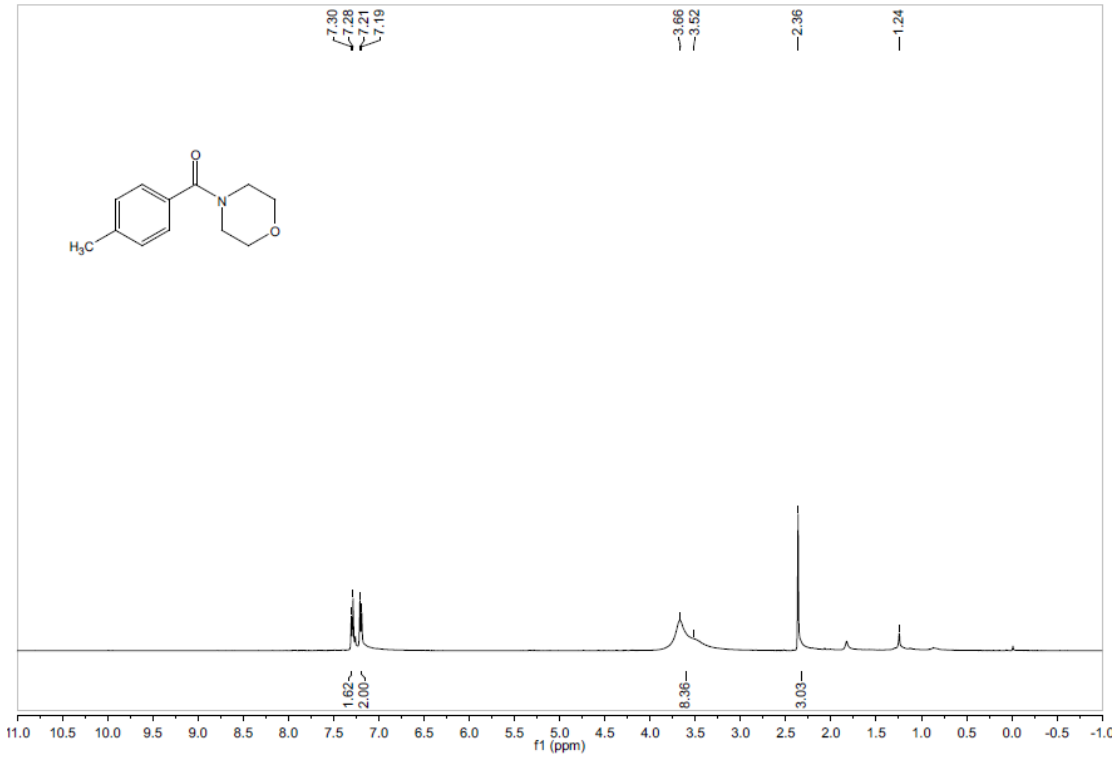
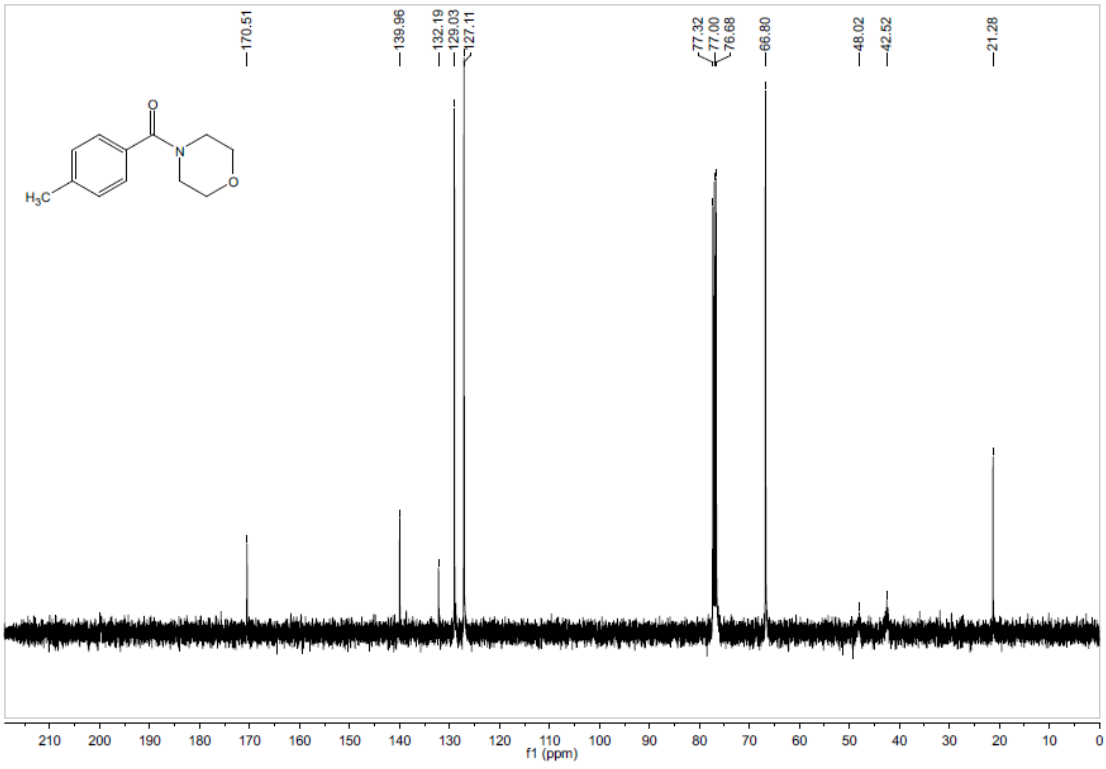
7.15
3.83
3.82
3.78
3.77
3.58
3.25
3.23
2.32
1.26
O
N
O
CH3
0.89
1.89
1.03
4.08
2.44
2.35
3.00
f1 (ppm)

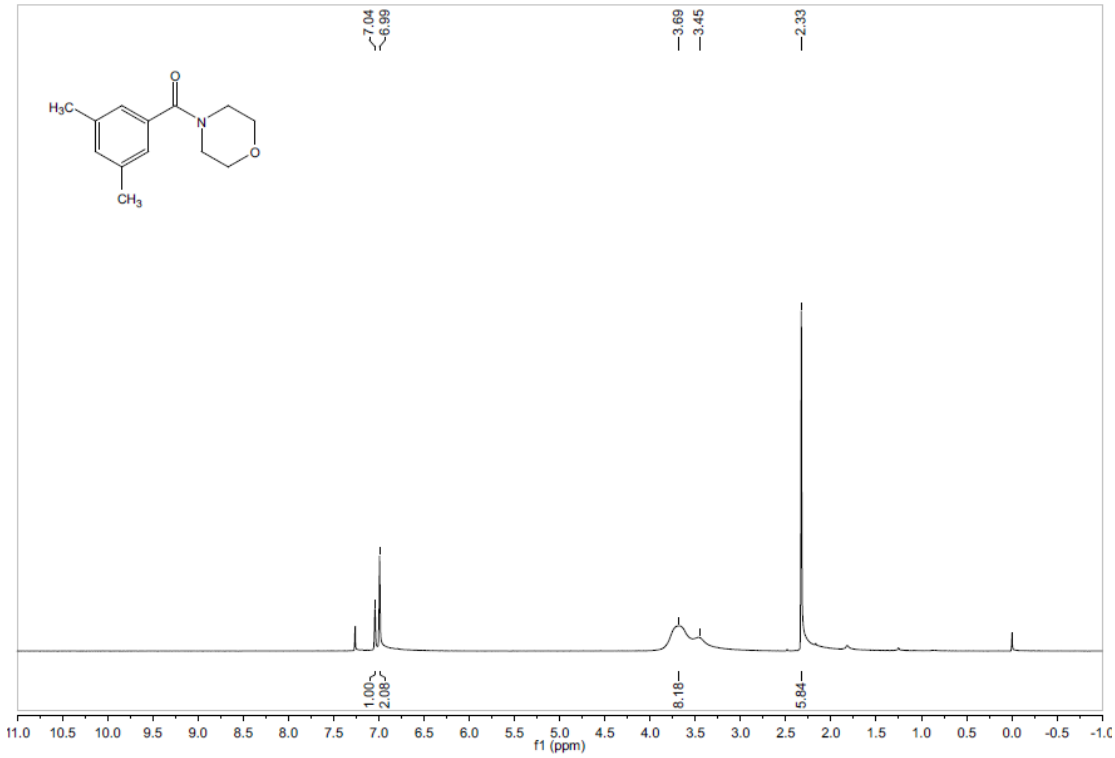

170.05
135.60
134.13
130.44
128.99
125.94
125.77
77.32
77.00
76.68
66.93
47.21
41.87
18.95
O
N
O
CH3
f1 (ppm)

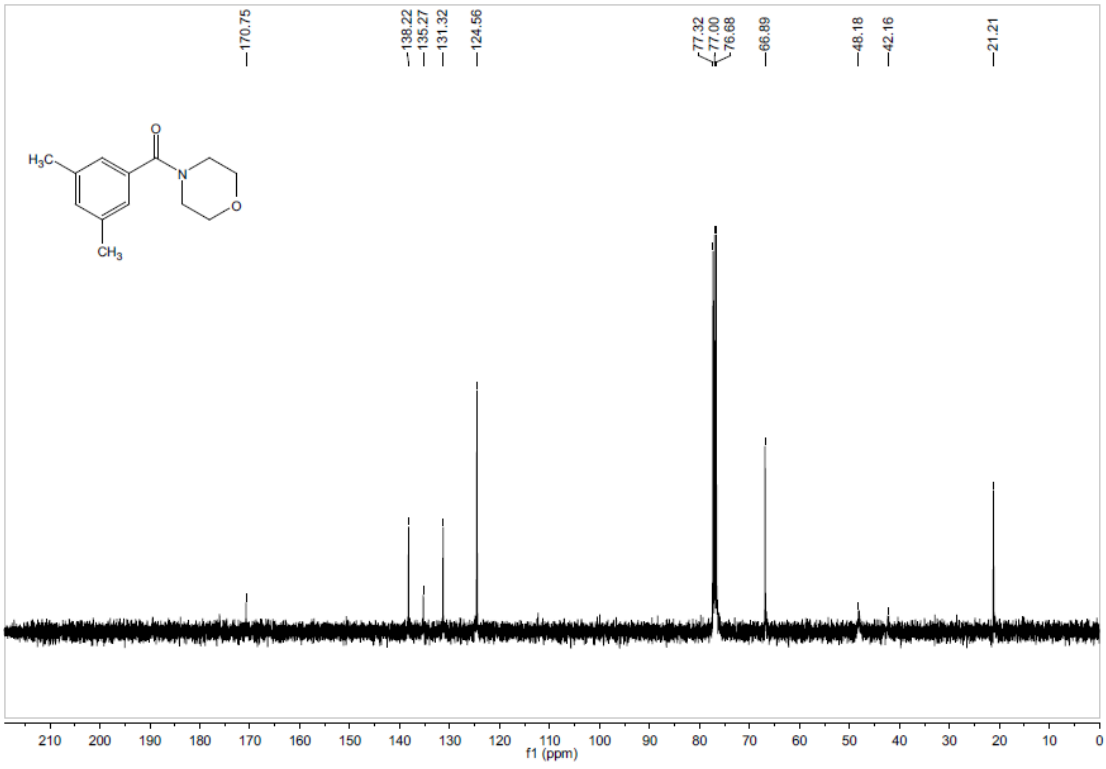

7.31
7.29
7.27
7.24
7.22
7.18
7.16
3.76
3.63
3.50
3.44
3.43
2.38
O
N
O
CH3
4.00
8.35
3.01
f1 (ppm)


170.58
138.46
135.26
127.64
123.91
77.32
77.00
76.68
66.86
48.13
42.48
21.30
O
N
O
CH3
f1 (ppm)


7.30
7.28
7.21
7.19
3.66
3.52
2.36
1.24
O
N
O
H3C
1.62
2.00
8.36
3.03
f1 (ppm)


170.51
139.96
132.19
129.03
127.11
77.32
77.00
76.68
66.80
48.02
42.52
21.28
O
N
O
H3C
f1 (ppm)


7.04
6.99
3.69
3.45
2.33
1.00
2.08
8.18
5.84
f1 (ppm)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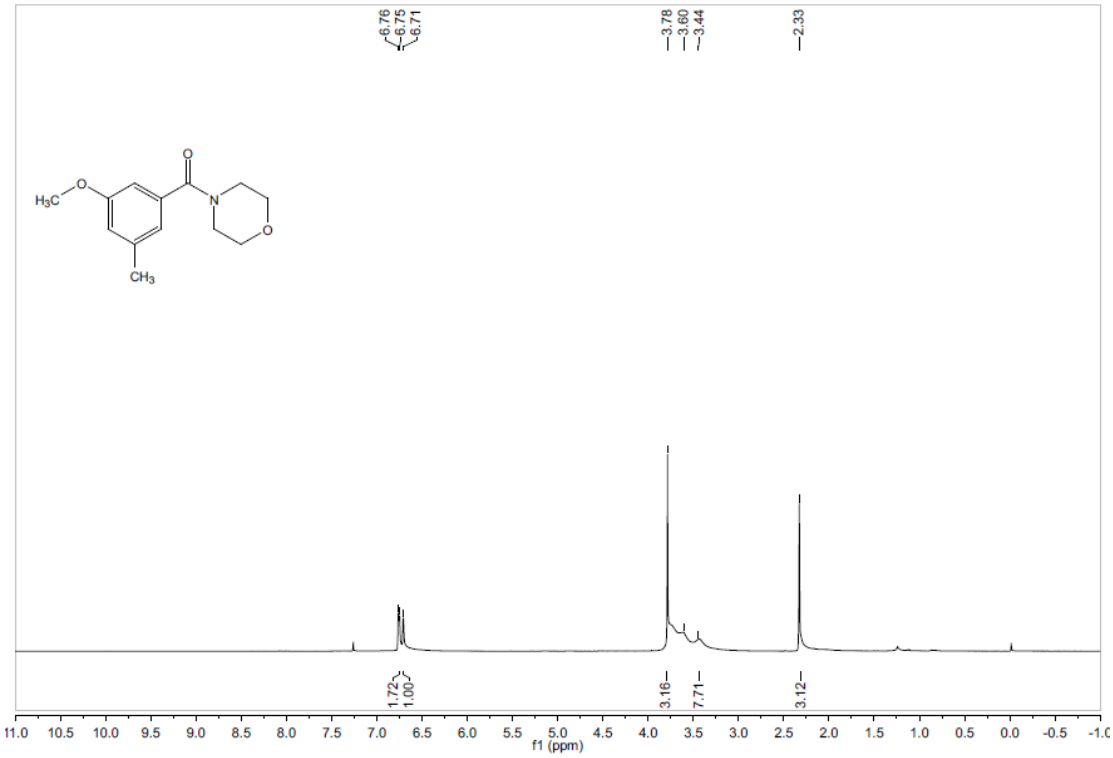
138.22
135.27
131.32
124.56
77.32
77.00
76.68
66.89
48.18
42.16
21.21
f1 (ppm)


6.76
6.75
6.71
3.78
3.60
3.44
2.33
O
N
O
H3C
O
CH3
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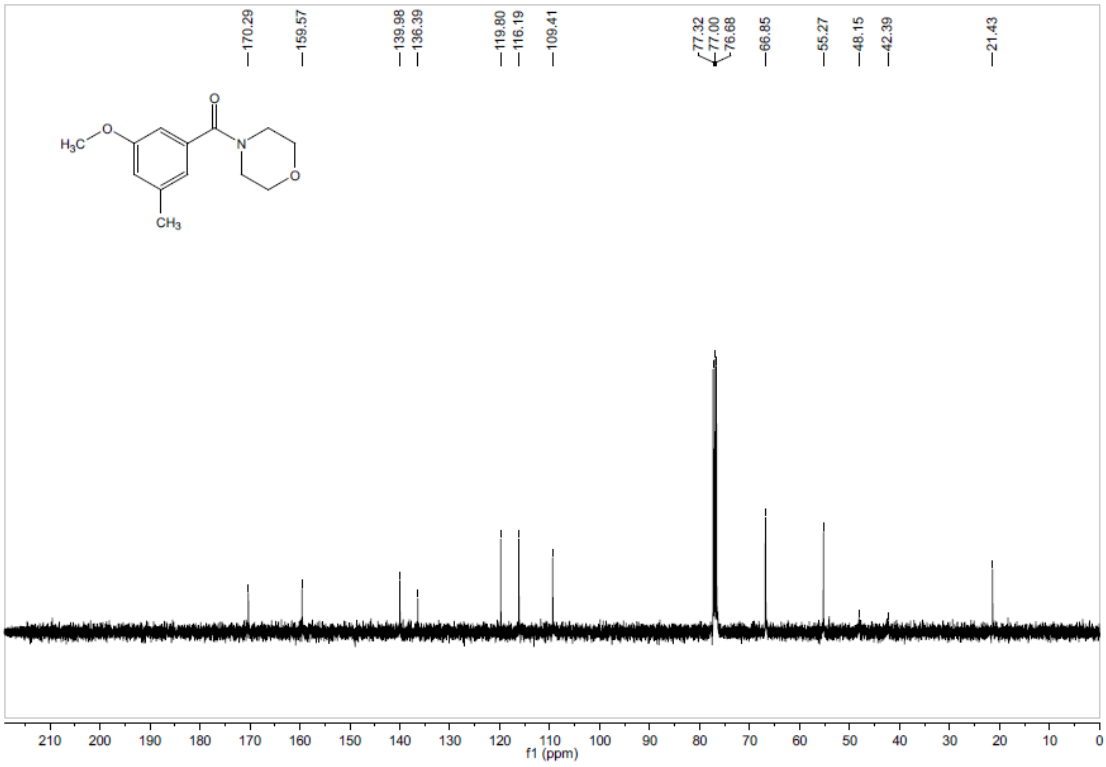
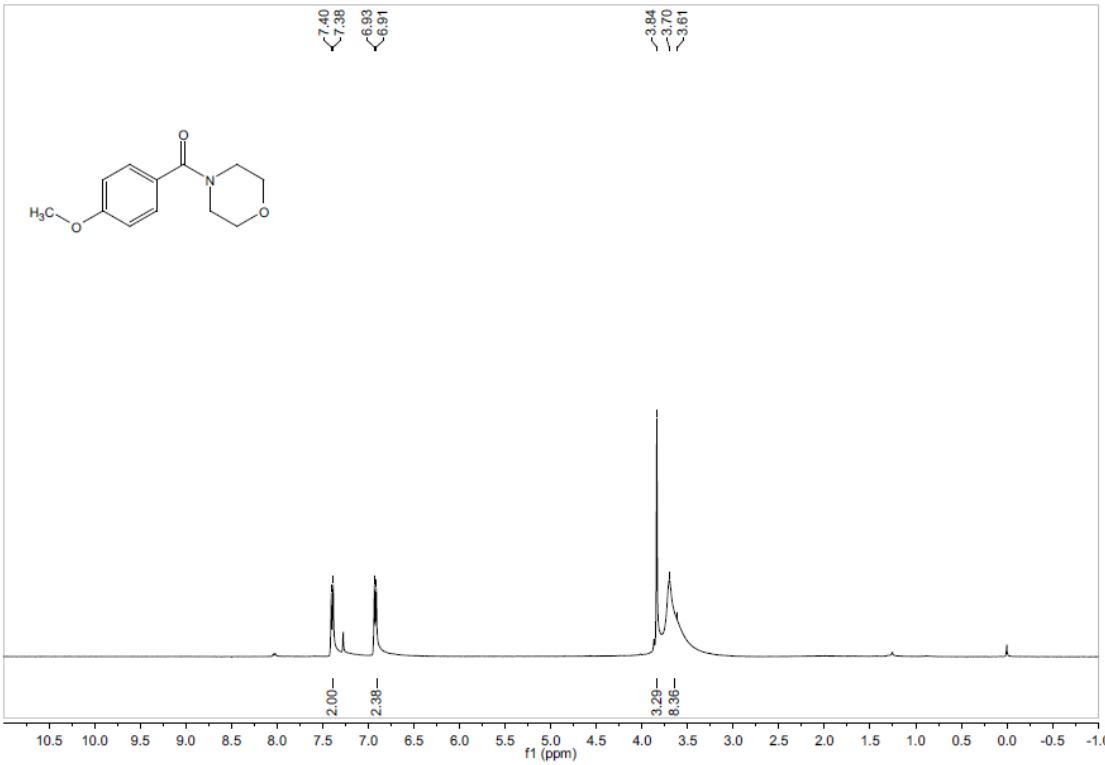
1.00
3.16
7.71
3.12
f1 (ppm)


170.29
159.57
139.98
136.39
119.80
116.19
109.41
77.32
77.00
76.68
66.85
55.27
48.15
42.39
21.43
O
N
O
H3C
O
CH3
f1 (ppm)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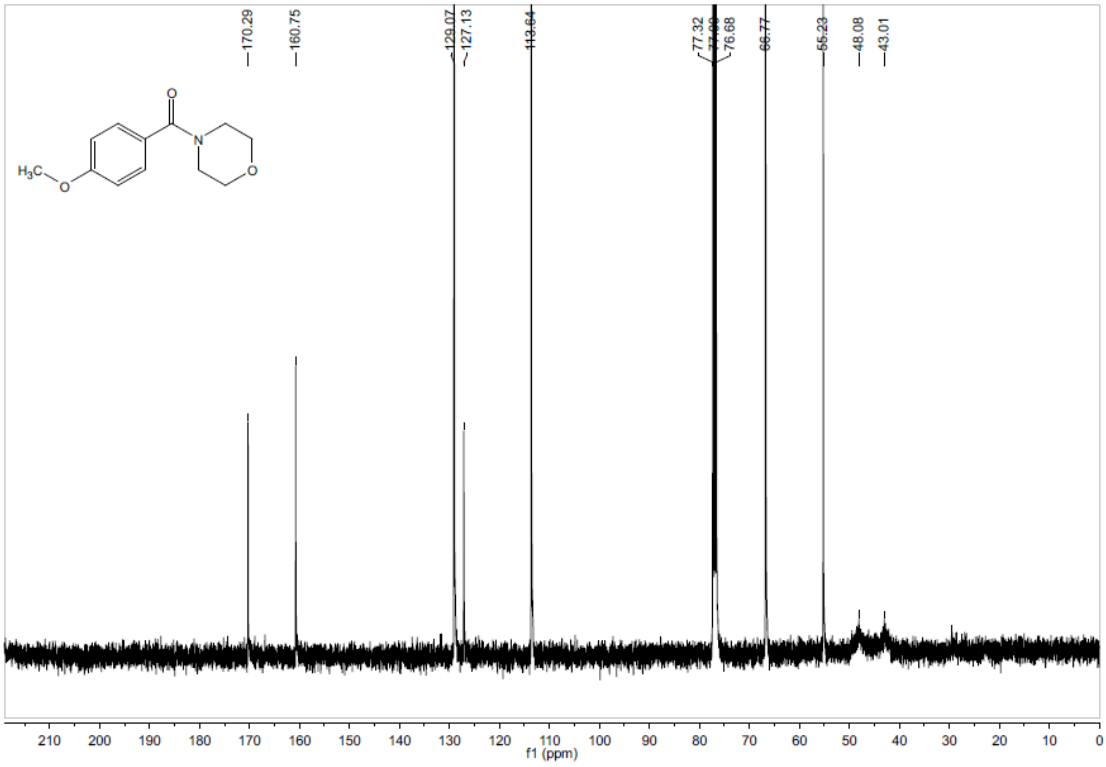
7.38
6.93
6.91
3.84
3.70
3.61
2.00
2.38
3.29
8.36
f1 (ppm)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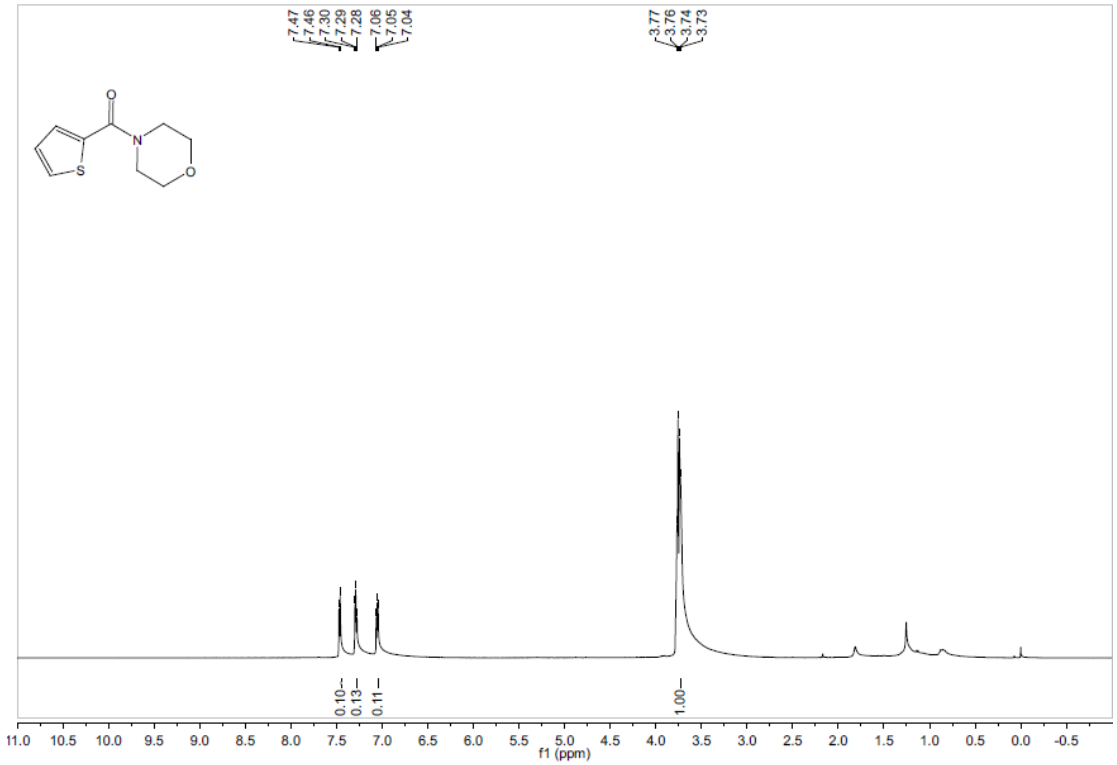
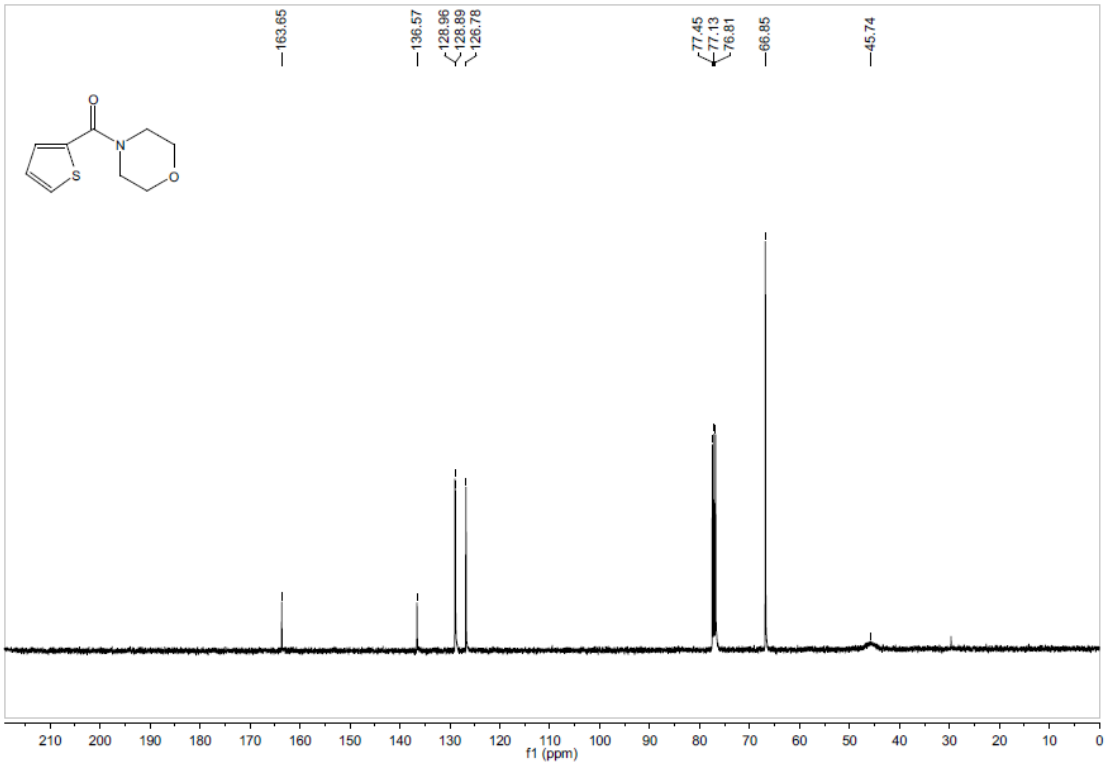
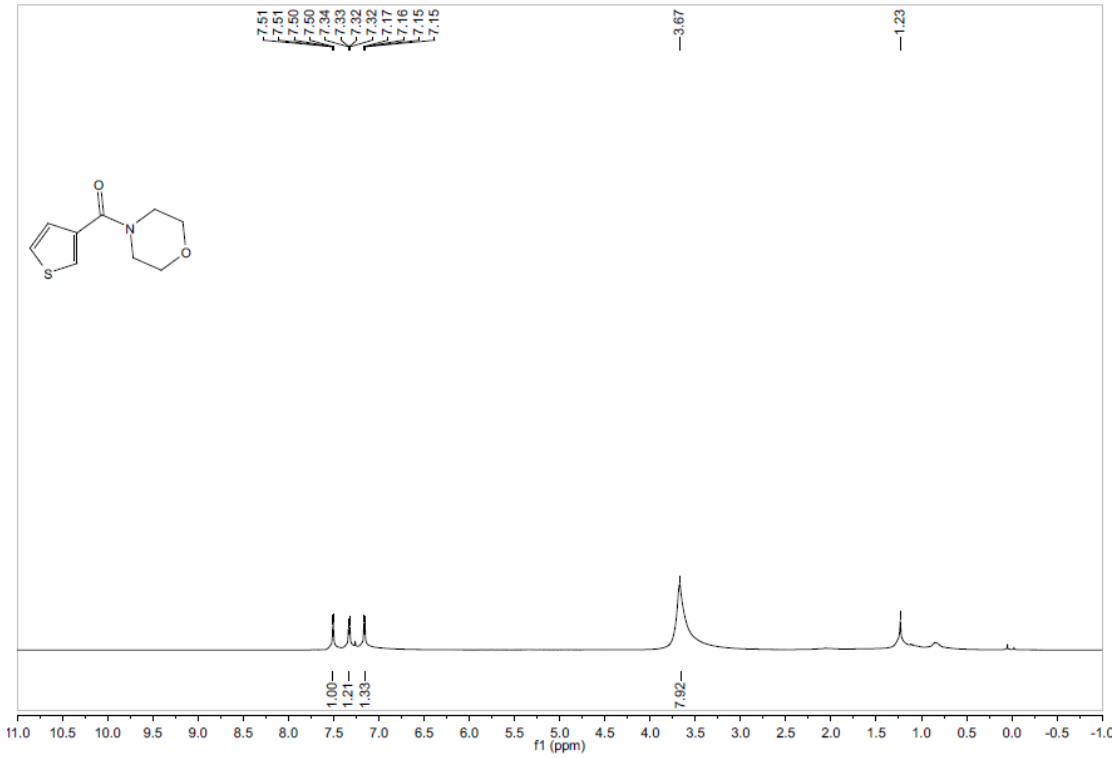
160.75
129.07
127.13
113.64
77.32
76.68
66.77
55.23
48.08
43.01
f1 (ppm)


7.47
7.46
7.30
7.29
7.28
7.06
7.05
7.04
3.77
3.76
3.74
3.73
O
N
S
O
0.10
0.13
0.11
1.00
f1 (ppm)


163.65
136.57
128.96
128.89
126.78
77.45
77.13
76.81
66.85
45.74
O
N
S
O
f1 (ppm)


7.51
7.51
7.50
7.50
7.34
7.33
7.32
7.32
7.17
7.16
7.15
7.15
3.67
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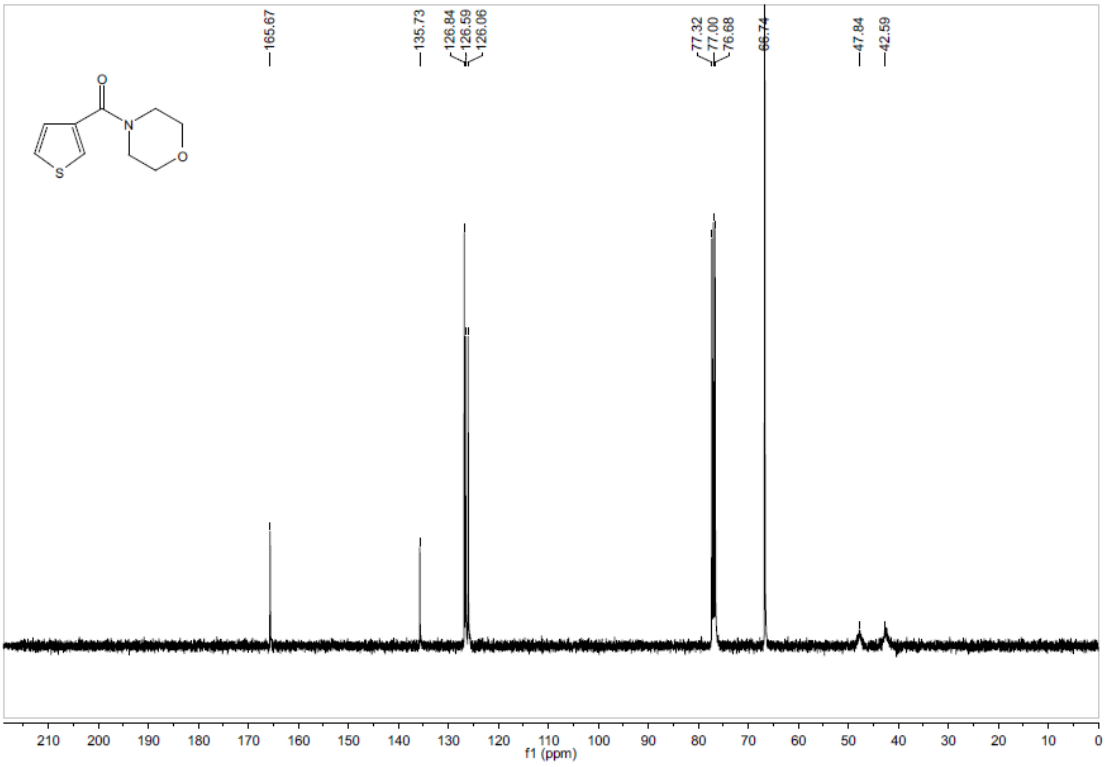
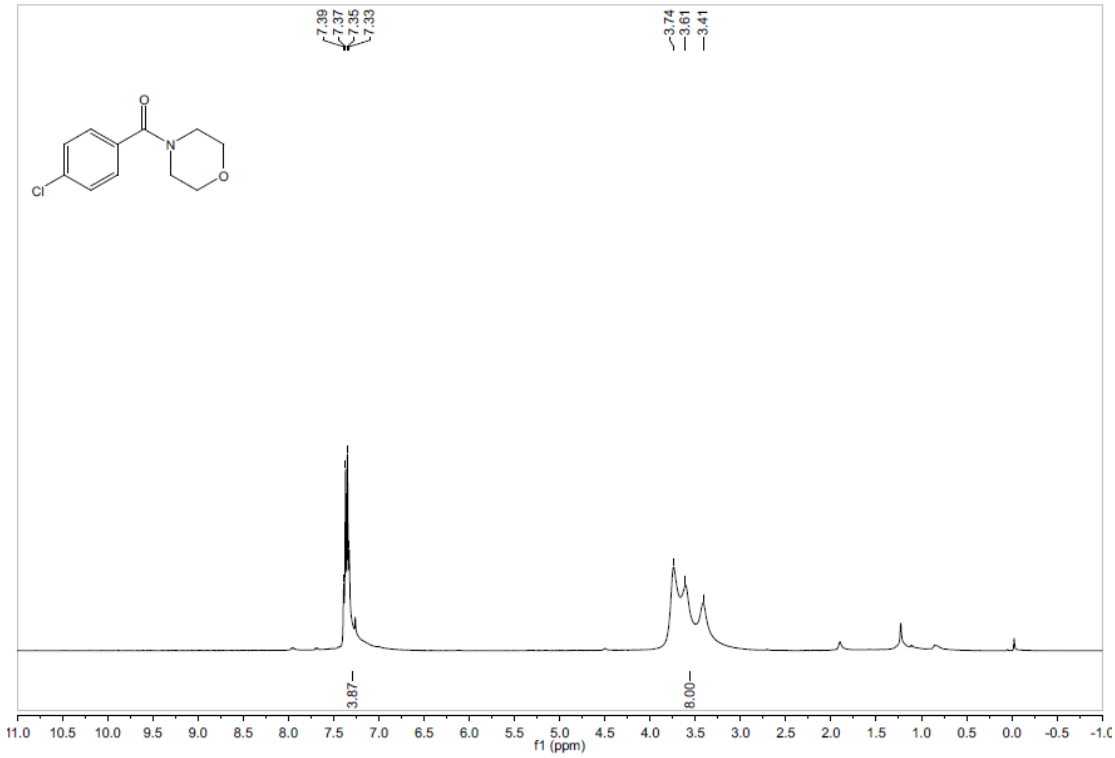
1.00
1.21
1.33
7.92
f1 (ppm)


165.67
135.73
126.84
126.59
126.06
77.32
77.00
76.68
66.74
47.84
42.59
f1 (ppm)


7.39
7.37
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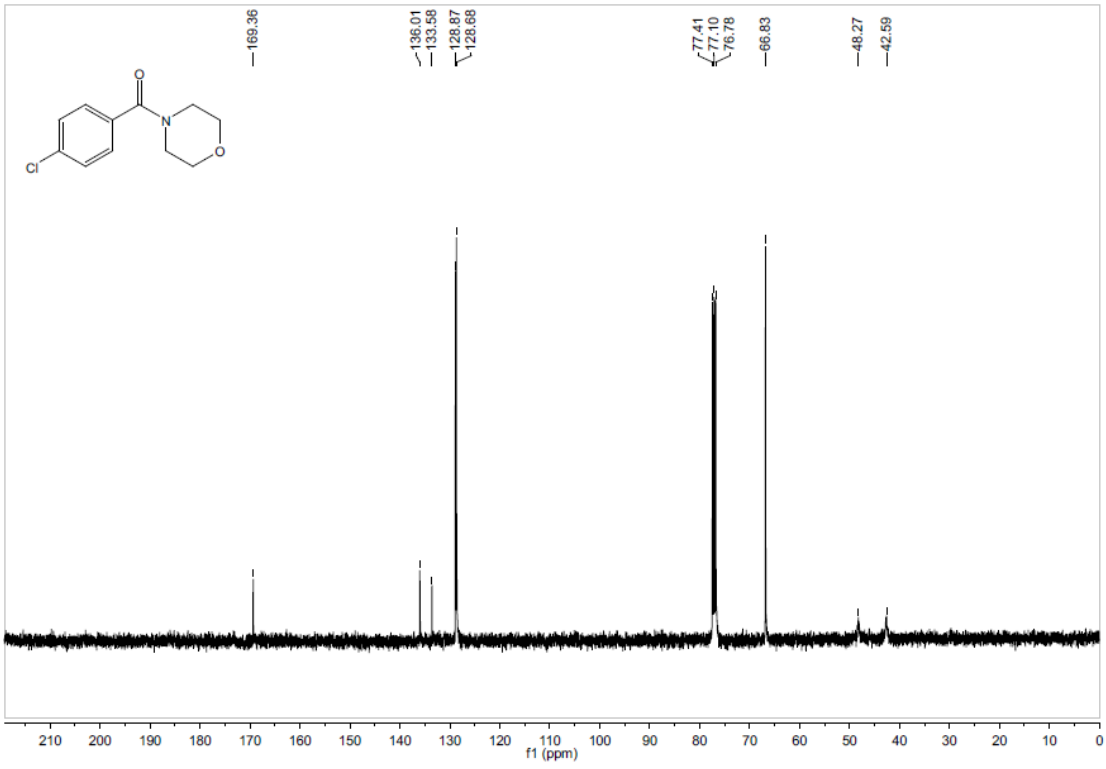
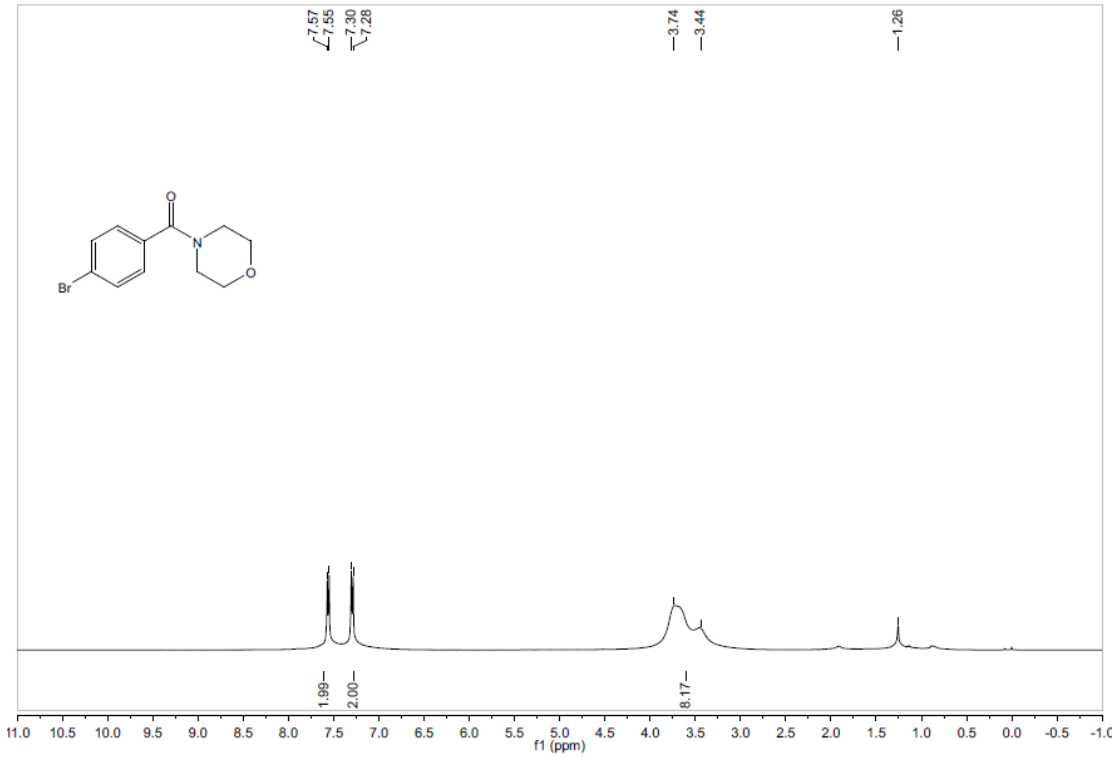
7.33
3.74
3.61
3.41
O
N
O
Cl
3.87
8.00
f1 (ppm)


169.36
136.01
133.58
128.87
128.68
77.41
77.10
76.78
66.83
48.27
42.59
O
N
O
Cl
f1 (ppm)


7.57
7.55
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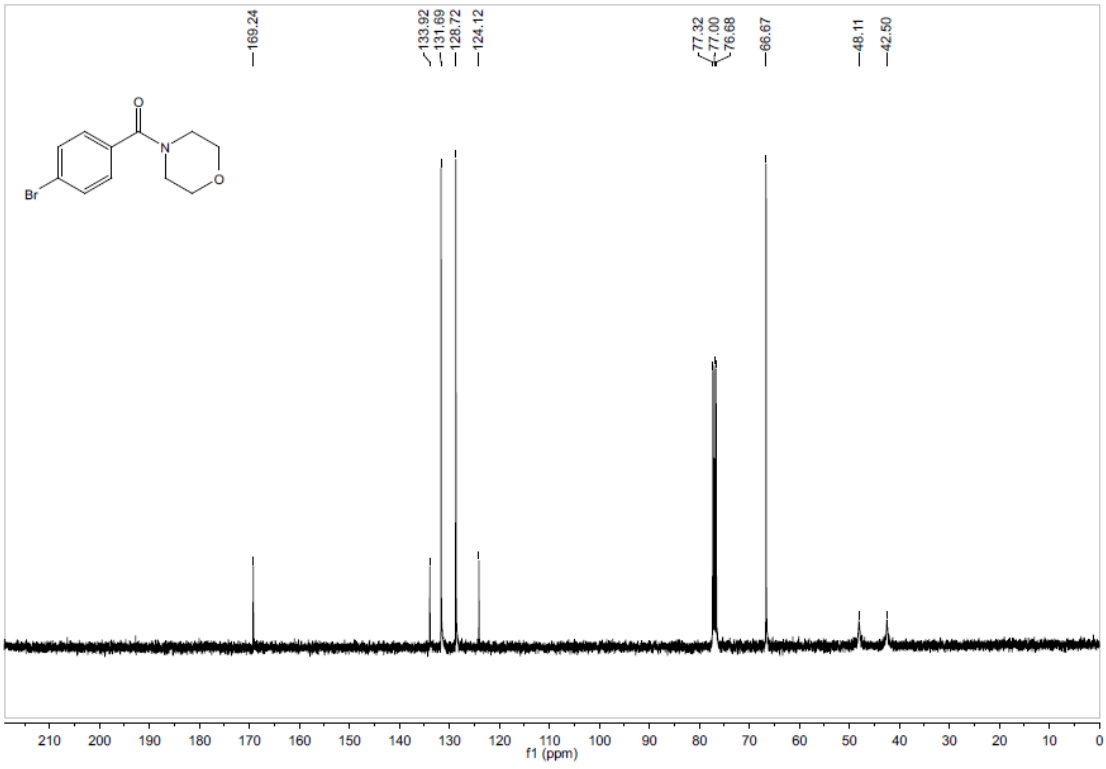
7.28
3.74
3.44
1.26
O
N
O
Br
1.99
2.00
8.17
f1 (ppm)


169.24
133.92
131.69
128.72
124.12
77.32
77.00
76.68
66.67
48.11
42.50
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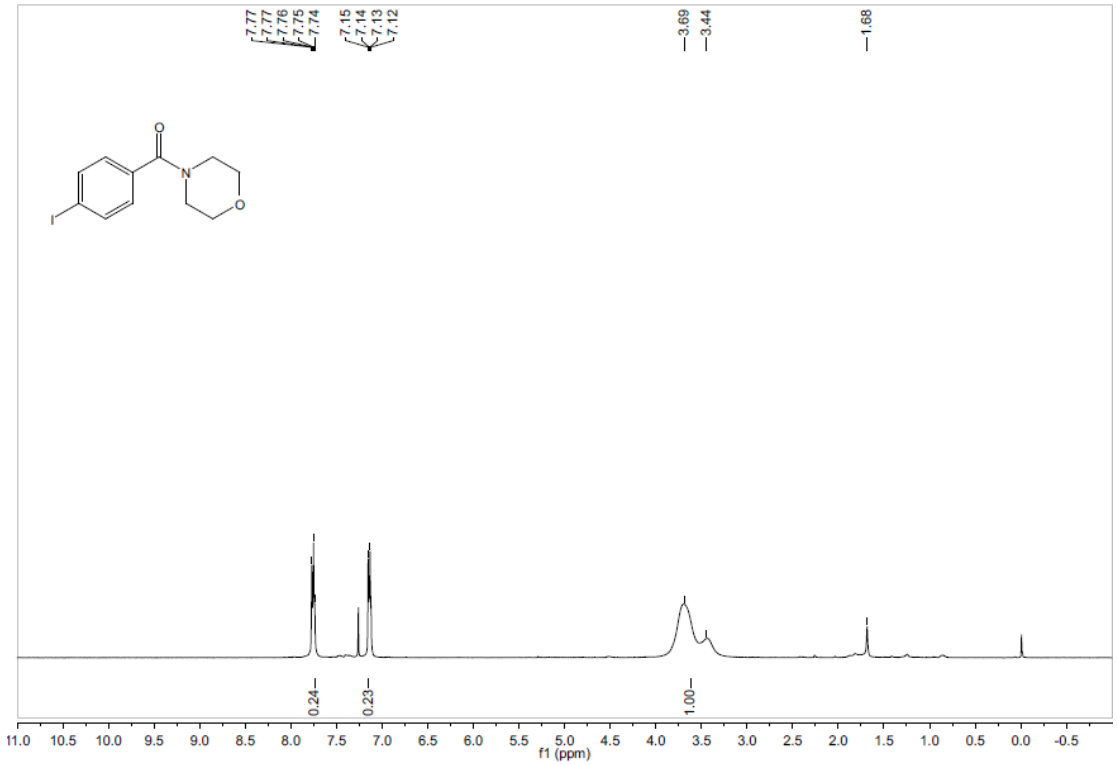
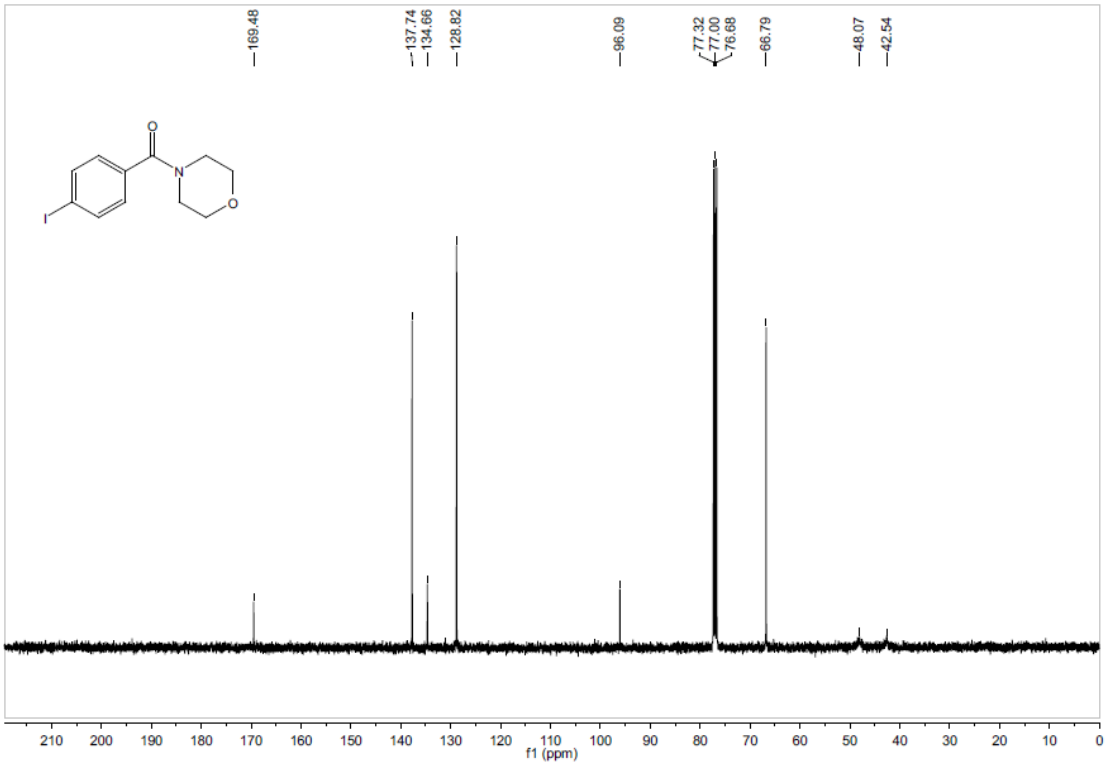
N
O
Br
f1 (ppm)


7.77
7.77
7.76
7.75
7.74
7.15
7.14
7.13
7.12
3.69
3.44
1.68
O
N
O
I
0.24
0.23
1.00
f1 (ppm)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128.82
96.09
77.32
77.00
76.68
66.79
48.07
42.54
O
N
O
I
f1 (ppm)