

Supporting Information

Visible light -induced photoredox catalyzed C-N coupling of amides with alcohols

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General Remarks

All chemicals were reagent grade, obtained from Merck and further they were used without purification.

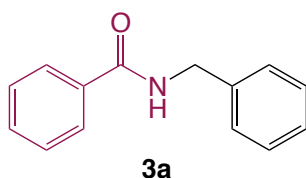
General experimental Procedure

In a tube furnished with a magnetic stirrer bar, benzamide (1 mmol), benzyl alcohol (1 mmol), 0.5 equivalent of KOH and eosin Y (2 mol%) were added to 3 mL of EtOH. The resulting mixture was stirred at room temperature for 6-10 h, under green LED irradiation to give the intermediate **I** (without isolation), further which was reduced by 0.5 equivalent of NaBH₄ to give the desired product (**3a-m**). To obtain the pure product **3a-m**, the crude product was purified by silica gel chromatography (100-200 mesh silica gel; EtOAc/Hexane). All of the products were identified by their spectral data and chemical analysis.

Supporting Information Summary

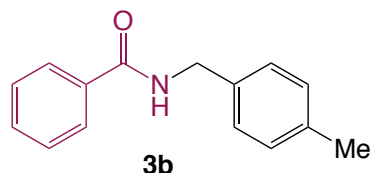
General experimental, general method for the synthesis of *N*-alkylated compounds, and characterization information of products. Melting points were determined by open glass capillary method and are uncorrected. All chemicals used were reagent grade and were used as received. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker AVANCE DPX (400 MHz and 100 MHz) FT spectrometer in CDCl₃ using TMS as an internal reference (chemical shift in δ ppm).

N-benzylbenzamide (**3a**)



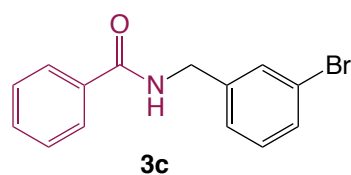
White solid, 96% yield. mp;180-181 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.79 (d, 2H), 7.50 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.4$ Hz, 2H), 7.37-7.35 (m = 4H), 7.32-7.28 (m, 1H), 6.44 (s, 1H), 4.65 (d, $J = 5.7$ Hz, 2H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 167.4, 138.2, 134.3, 131.5, 128.7, 128.5, 127.9, 127.6, 126.9, 44.1.

***N*-(4-methylbenzyl)benzamide (3b)**



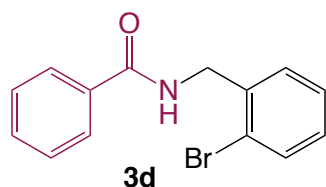
White solid, 95% yield. mp;196-197 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.78 (d, $J = 7.3$ Hz, 2H), 7.49 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.6$ Hz, 2H), 7.25 (d, $J = 4.3$ Hz, 2H), 7.16 (d, $J = 4.3$ Hz, 2H), 6.39 (s, 1H), 4.60 (d, $J = 5.7$ Hz, 2H), 2.34 (s, 3H). C NMR (CDCl_3 , 100 MHz): δ 167.2, 159.2, 134.5, 131.5, 130.2, 129.3, 128.6, 126.9, 114.2, 55.3, 43.7

***N*-(3-bromobenzyl)benzamide (3c)**



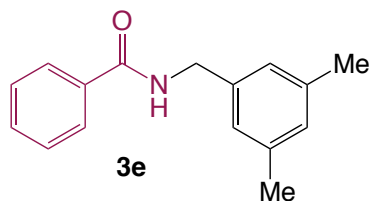
White solid, 97% yield. m.p: 241-242 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.83 – 7.74 (m, 2H), 7.55 – 7.47 (m, 2H), 7.47 – 7.38 (m, 3H), 7.32 – 7.26 (m, 1H), 7.21 (t, $J = 7.7$ Hz, 1H), 6.55 (s, 1H), 4.61 (d, $J = 5.8$ Hz, 2H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 167.5, 140.7, 134.2, 131.9, 130.9, 130.8, 130.5, 128.8, 127.1, 126.6, 122.9, 43.6.

***N*-(2-bromobenzyl)benzamide (3d)**



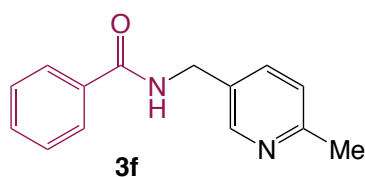
White solid, 82% yield. m.p: 241-242 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.84 – 7.74 (m, 2H), 7.57 (dd, $J = 7.9, 1.3$ Hz, 1H), 7.54 – 7.39 (m, 4H), 7.29 (td, $J = 7.5, 1.3$ Hz, 1H), 7.16 (td, $J = 7.7, 1.8$ Hz, 1H), 6.69 (s, 1H), 4.71 (d, $J = 6.0$ Hz, 2H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 167.4, 137.4, 134.4, 133.0, 131.8, 130.8, 129.4, 128.7, 127.9, 127.1, 124.0, 44.5.

***N*-(3,5-dimethylbenzyl)benzamide (3e)**



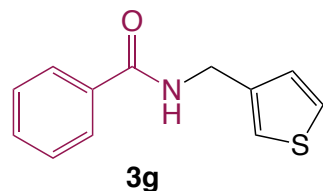
White solid, 98% yield. m.p: 228-229 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.84 – 7.77 (m, 2H), 7.53 – 7.46 (m, 1H), 7.42 (ddt, J = 8.3, 6.5, 1.3 Hz, 2H), 6.97 (d, J = 1.6 Hz, 2H), 6.94 (s, 1H), 6.51 (s, 1H), 4.56 (d, J = 5.6 Hz, 2H), 2.31 (d, J = 0.8 Hz, 6H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 167.4, 138.5, 138.2, 134.6, 131.6, 129.3, 128.7, 127.1, 125.9, 44.2, 21.4.

***N*-((6-methylpyridin-3-yl)methyl)benzamide (3f)**



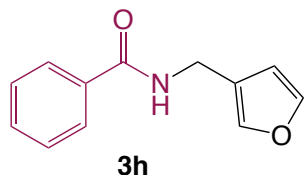
White solid, 90% yield. m.p: 258-259 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 8.50 (d, J = 2.3 Hz, 1H), 7.82 – 7.75 (m, 2H), 7.64 (dd, J = 8.0, 2.3 Hz, 1H), 7.54 – 7.48 (m, 1H), 7.44 (ddt, J = 8.2, 6.6, 1.3 Hz, 2H), 7.16 (d, J = 7.9 Hz, 1H), 6.49 (s, 1H), 4.63 (d, J = 5.8 Hz, 2H), 2.56 (s, 3H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 167.7, 157.7, 148.5, 136.5, 134.2, 131.8, 131.2, 128.8, 127.1, 123.4, 41.3, 24.1.

***N*-(thiophen-3-ylmethyl)benzamide (3g)**



White solid, 97% yield. m.p: 236-237 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.82 – 7.73 (m, 2H), 7.52 – 7.45 (m, 1H), 7.45 – 7.36 (m, 2H), 7.30 (dd, J = 5.0, 2.9 Hz, 1H), 7.19 (qd, J = 3.0, 2.0, 1.3 Hz, 1H), 7.07 (dd, J = 4.9, 1.4 Hz, 1H), 6.60 (s, 1H), 4.62 (d, J = 5.4 Hz, 2H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 167.4, 139.1, 134.4, 131.7, 128.7, 127.5, 127.08, 127.07, 126.59, 126.57, 122.6, 77.5, 77.2, 76.8, 39.4.

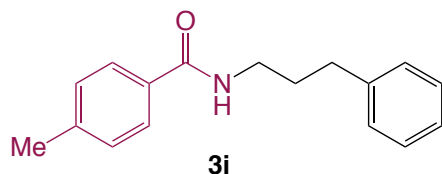
***N*-(furan-3-ylmethyl)benzamide (3h)**



Brown solid, 63% yield. m.p: 151-153 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.80 – 7.75 (m, 2H), 7.54 – 7.47 (m, 1H), 7.47 – 7.39 (m, 4H), 6.43 (dd, J = 1.9, 0.9 Hz, 1H), 6.24 (s, 1H), 4.50 (dd, J = 5.6, 0.9 Hz, 2H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 167.5, 143.8, 140.5, 134.5, 131.7, 128.8, 127.0, 122.3, 110.5,

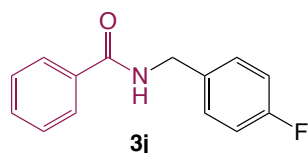
35.2.

4-methyl-N-(3-phenylpropyl)benzamide (3i)



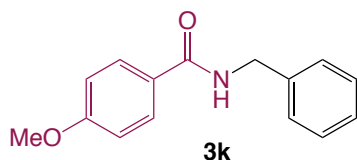
Yellow solid, 72% yield. m.p: 240-242 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.63 – 7.52 (m, 2H), 7.33 – 7.27 (m, 3H), 7.25 – 7.16 (m, 5H), 6.06 (s, 1H), 3.50 (td, J = 7.1, 5.8 Hz, 2H), 2.73 (t, J = 7.5 Hz, 2H), 2.38 (s, 4H), 2.03 – 1.88 (m, 2H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 167.5, 141.8, 141.7, 132.0, 129.3, 128.7, 128.5, 126.9, 126.2, 39.9, 33.7, 31.3, 21.6.

N-(4-fluorobenzyl)benzamide (3j)



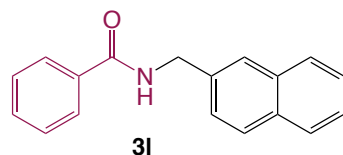
White solid, 96% yield. m.p: 196-198 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 8.08 – 8.04 (s, 1H), 7.9 – 7.8 (d, 2H), 7.79-7.50 (t, 1H), 7.49-7.41 (dd, J = 5.6 Hz, 2H), 7.40-7.35 (d, J = 5.6 Hz, 2H), 7.28-7.27 (d, J = 5.6 Hz, 2H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 168.4, 134.65, 131.71, 128.68, 127.91, 40.50-39.25.

N-benzyl-4-methoxybenzamide (3k)



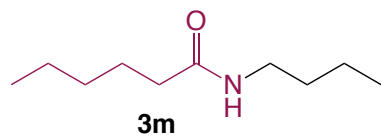
White solid, 97% yield. m.p: 226-228 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.03 (s, 1H), 7.0-6.98 (d, 2H), 6.06-6.04 (d, 2H), 2.98 (s, J = 5.6 Hz, 2H), 2.09 (s, J = 5.6 Hz, 3H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 168.4, 161.65, 128.93, 112.9, 77.25-76.74, 54.77, 39.9-38.9.

N-(naphthalen-2-ylmethyl)benzamide (3l)



White solid, 97% yield. m.p: 256-258 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.89 – 7.74 (m, 6H), 7.56 – 7.39 (m, 6H), 6.51 (s, 1H), 4.81 (d, J = 5.6 Hz, 2H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 167.5, 135.7, 134.5, 133.5, 133.0, 131.7, 128.8, 128.8, 127.9, 127.8, 127.1, 126.7, 126.5, 126.2, 126.1, 44.4.

***N*-butylhexanamide (3m)**



Colourless oil, 35% yield. ^1H NMR (400 MHz, CDCl_3): δ = 5.79 (br s, 1H), 3.20 (q, J = 6.8 Hz, 2H), 2.12 (t, J = 7.6 Hz, 2H), 1.62-1.55 (m, 2H), 1.48-1.40 (m, 2H), 1.33-1.21 (m, 6H), 0.90-0.83 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ = 173.14, 39.09, 36.73, 31.65, 31.40, 25.46, 22.31, 19.98, 13.83, 13.64.

^1H and ^{13}C NMR spectra of compounds

Figure S1a: ^1H NMR (400 MHz, CDCl_3) of *N*-benzylbenzamide (**3a**)

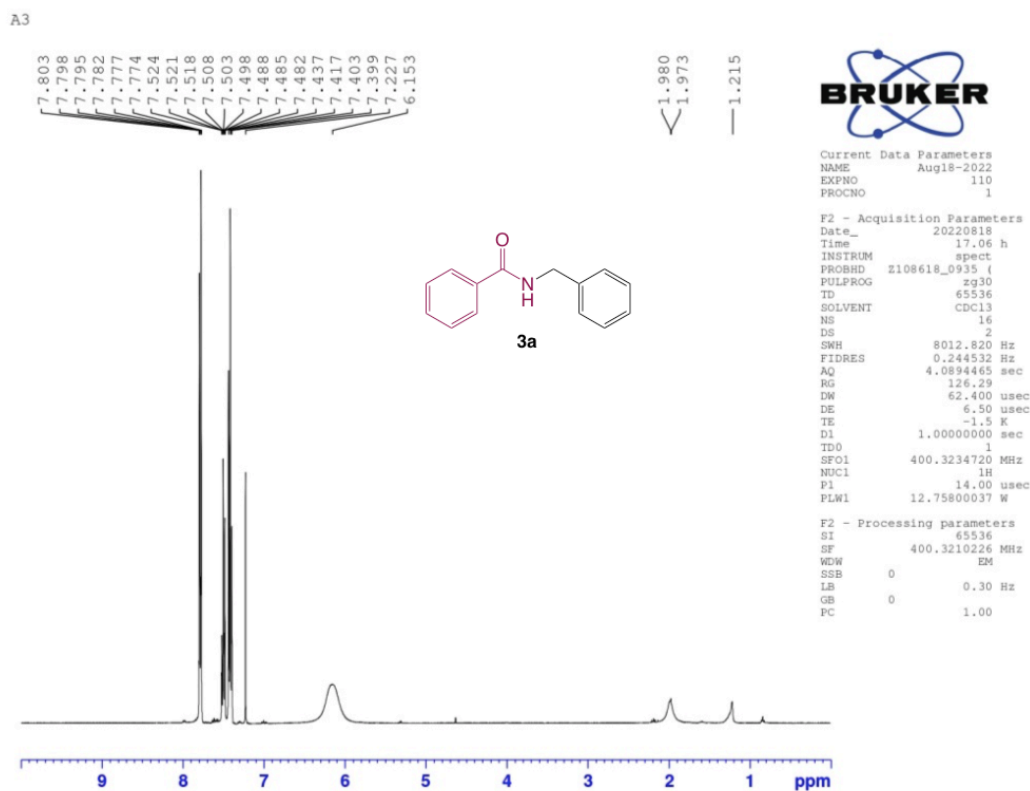


Figure S1b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-benzylbenzamide (**3a**)

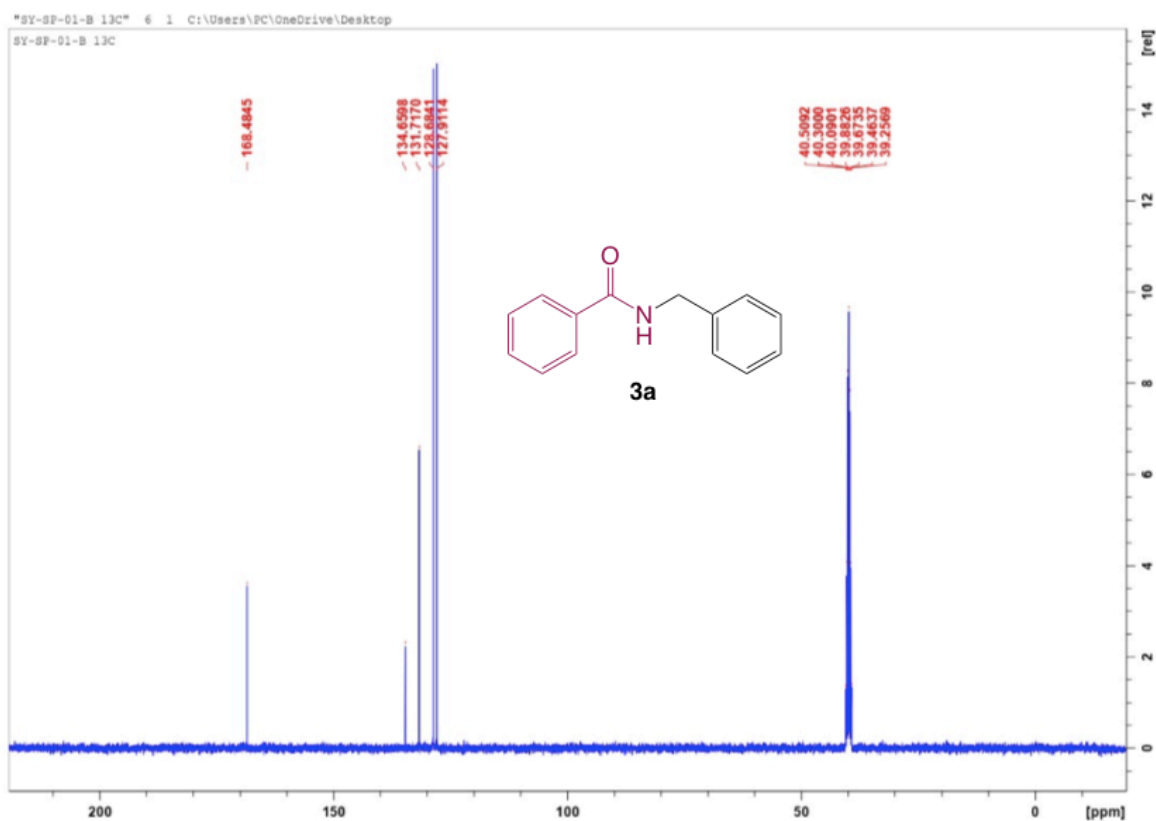


Figure S2a: ^1H NMR (400 MHz, CDCl_3) of *N*-(4-methylbenzyl)benzamide (**3b**)

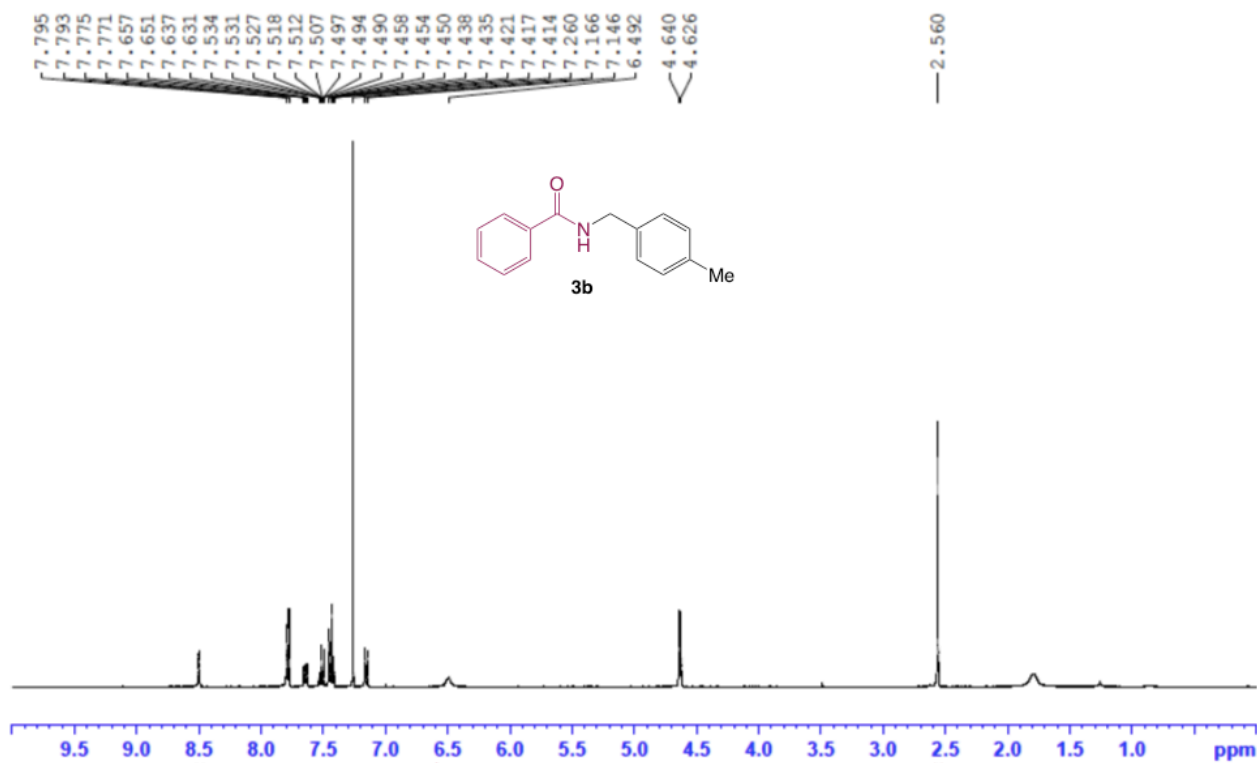


Figure S2b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-(4-methylbenzyl)benzamide (**3b**)

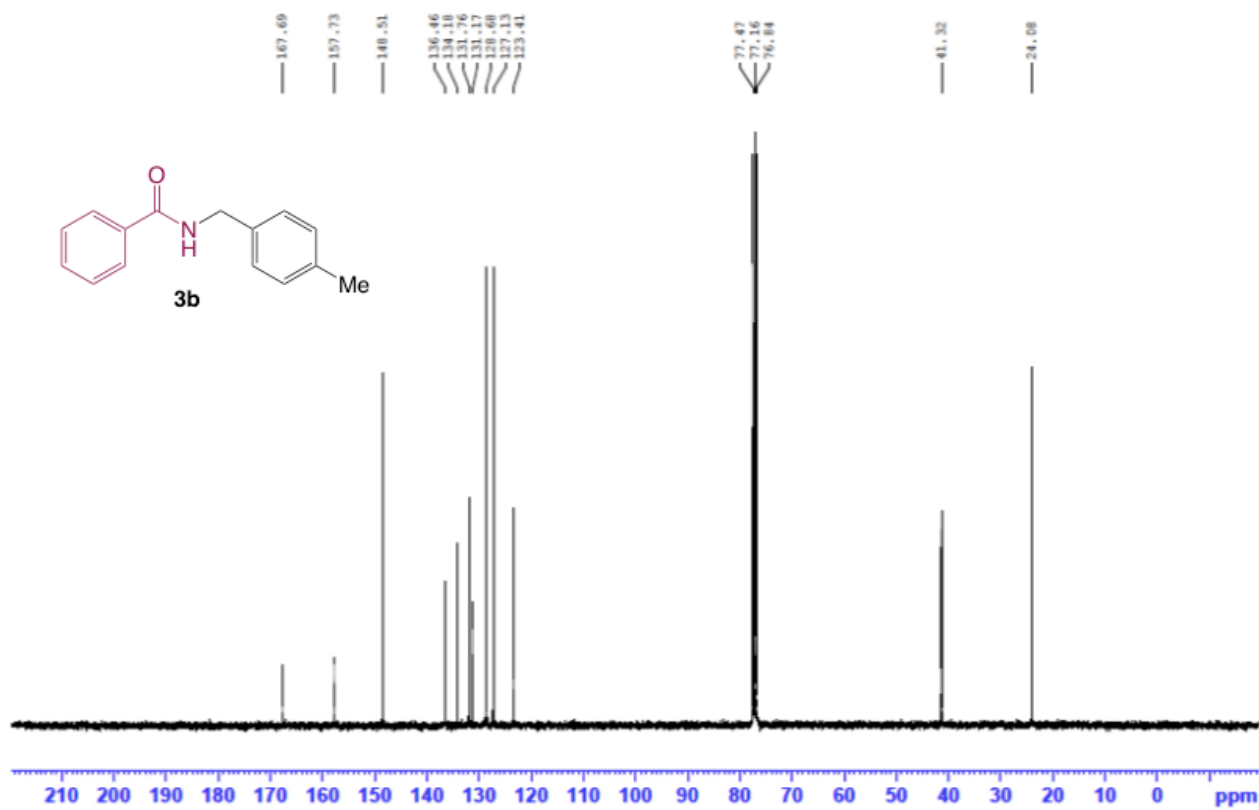


Figure S3a: ^1H NMR (400 MHz, CDCl_3) of *N*-(3-bromobenzyl)benzamide (**3c**)

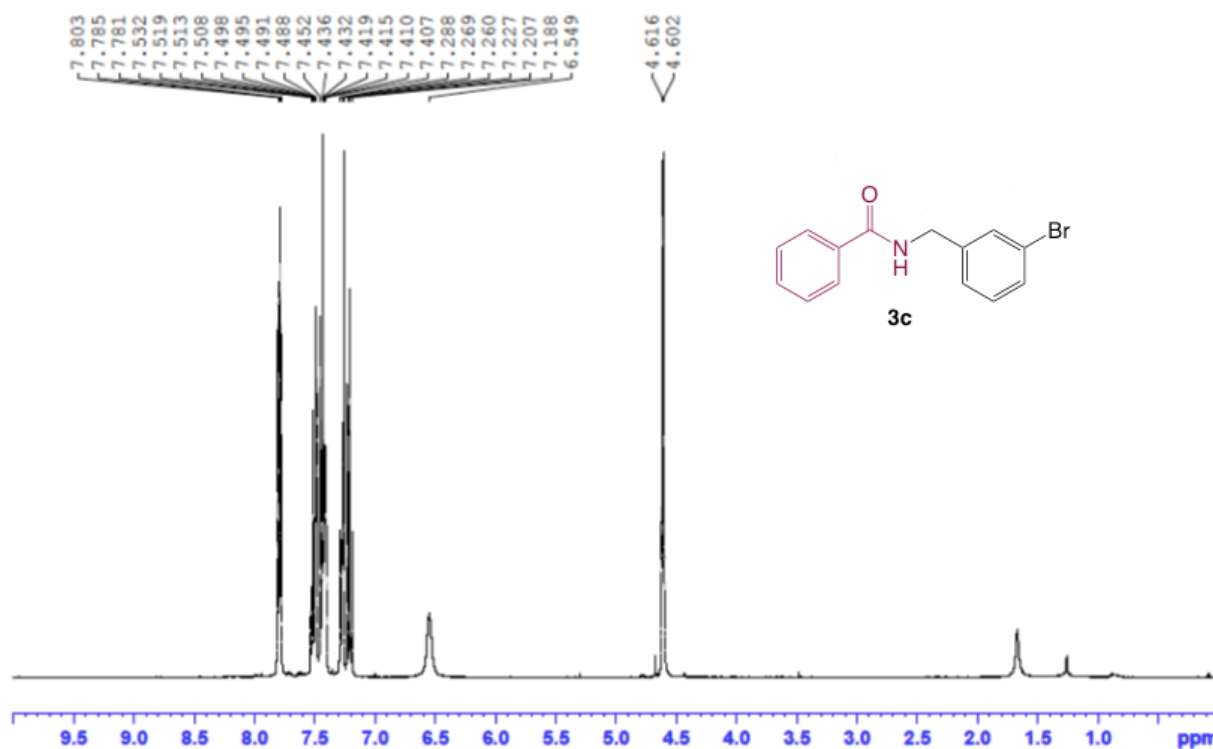


Figure S3b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-(3-bromobenzyl)benzamide (**3c**)

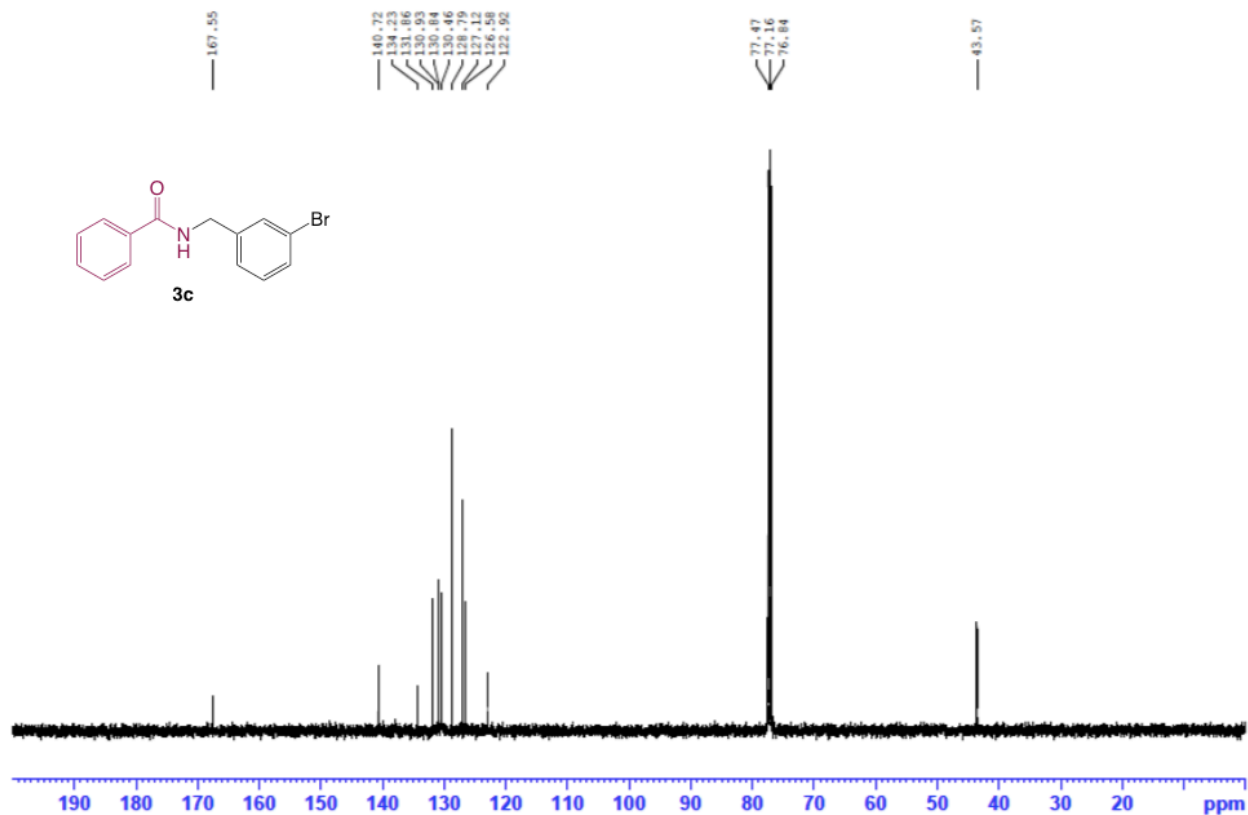


Figure S4a: ^1H NMR (400 MHz, CDCl_3) of *N*-(2-bromobenzyl)benzamide (**3d**)

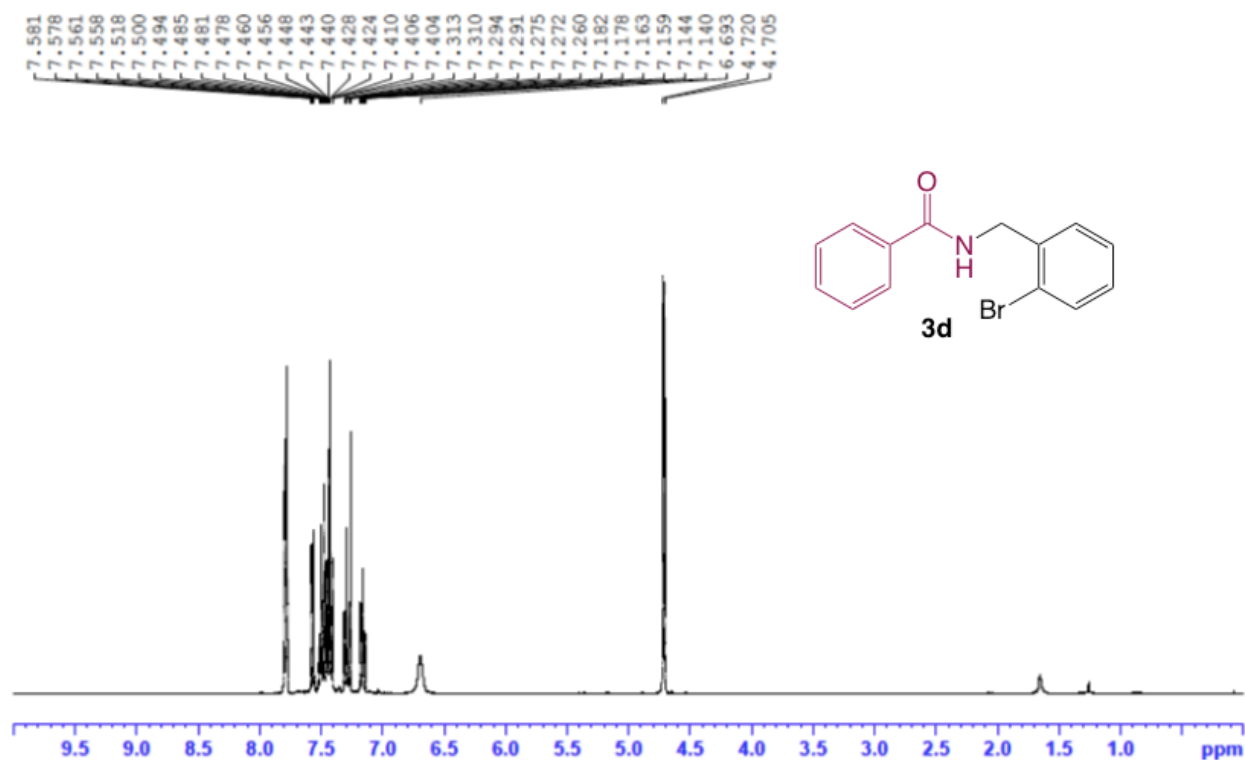


Figure S4b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-(2-bromobenzyl)benzamide (**3d**)

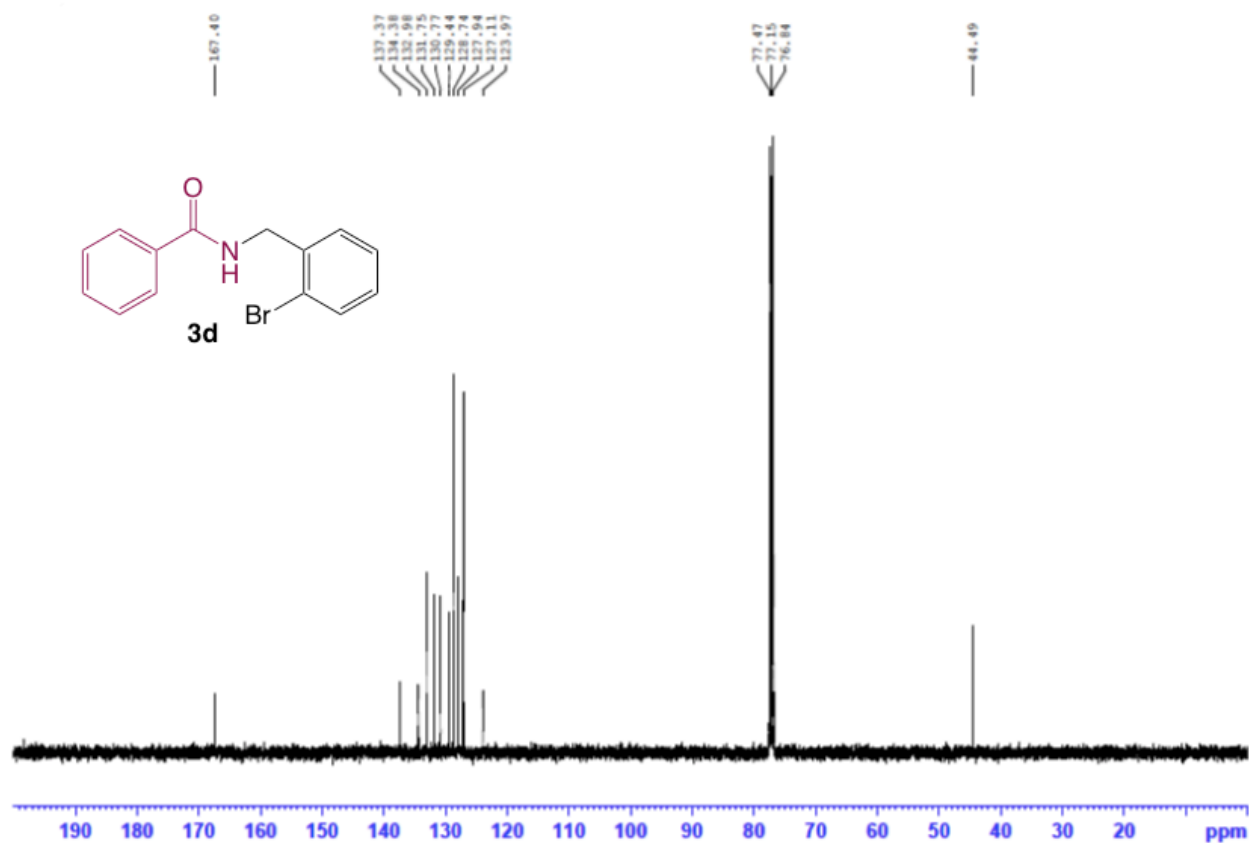


Figure S5a: ^1H NMR (400 MHz, CDCl_3) of *N*-(3,5-dimethylbenzyl)benzamide (**3e**)

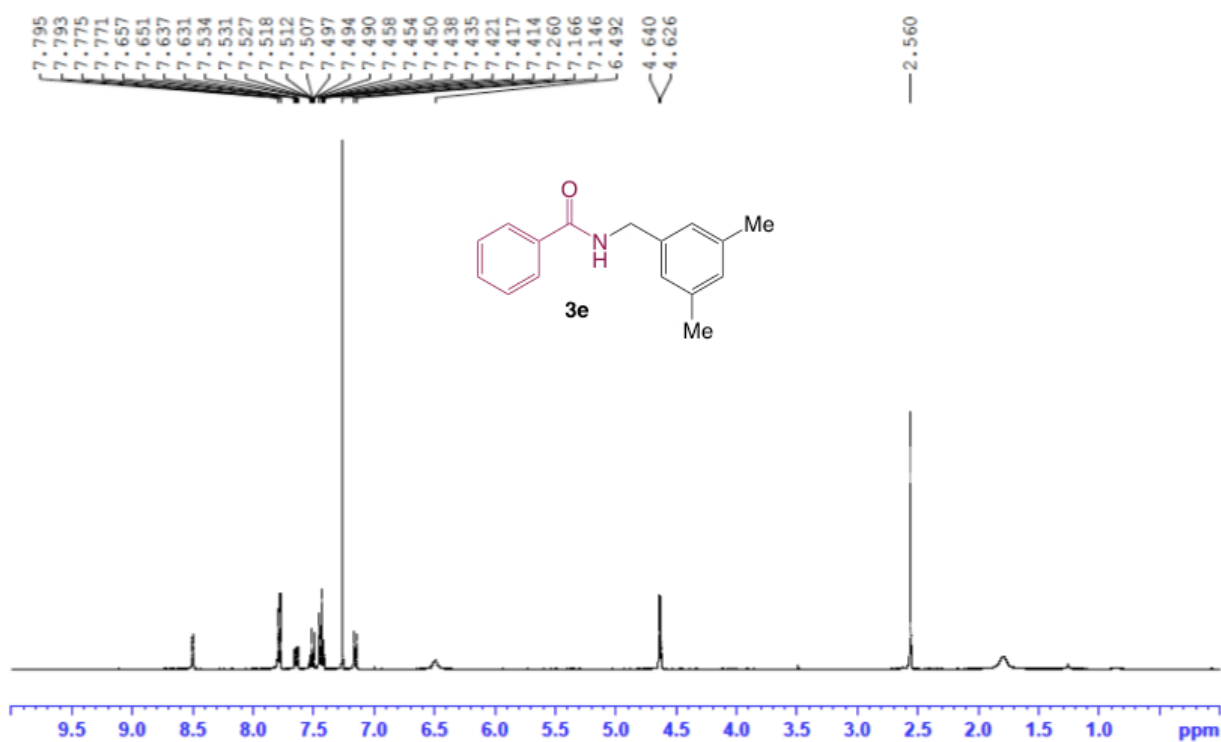


Figure S5b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-(3,5-dimethylbenzyl)benzamide (**3e**)

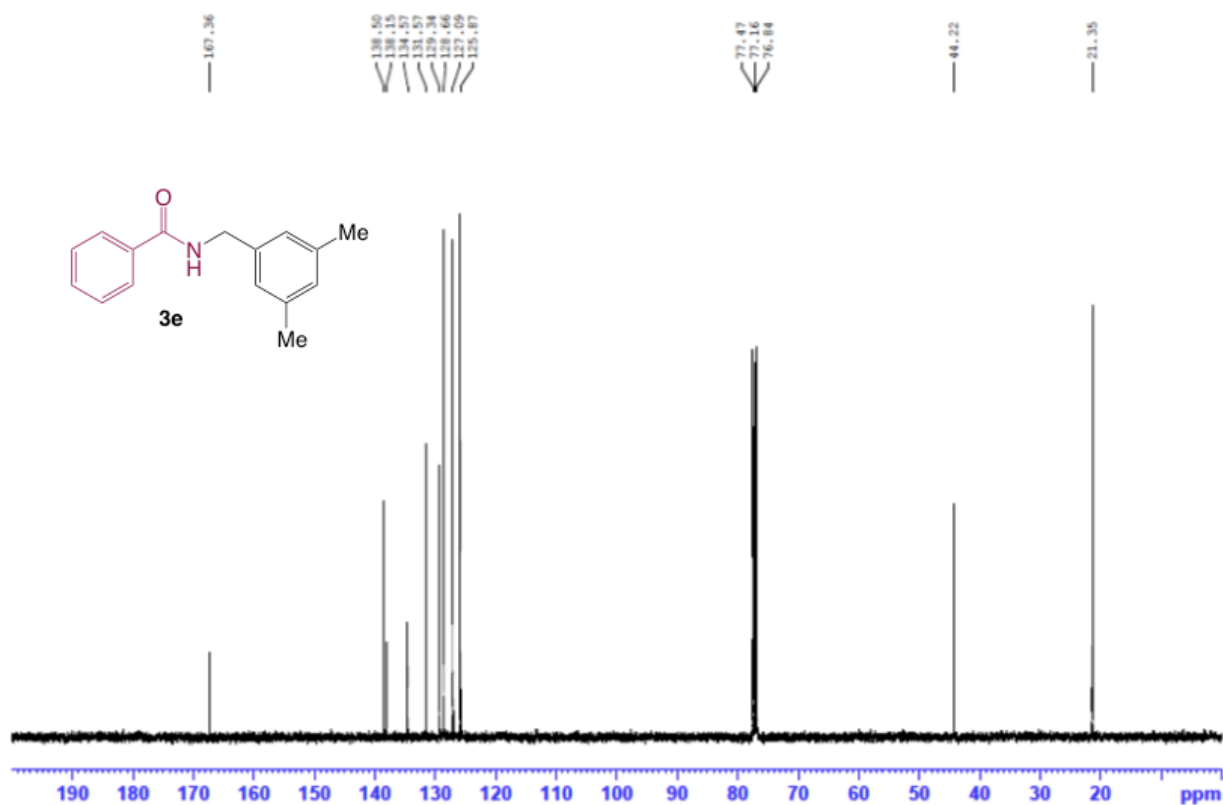


Figure S6a: ^1H NMR (400 MHz, CDCl_3) of *N*-((6-methylpyridin-3-yl)methyl)benzamide (**3f**)

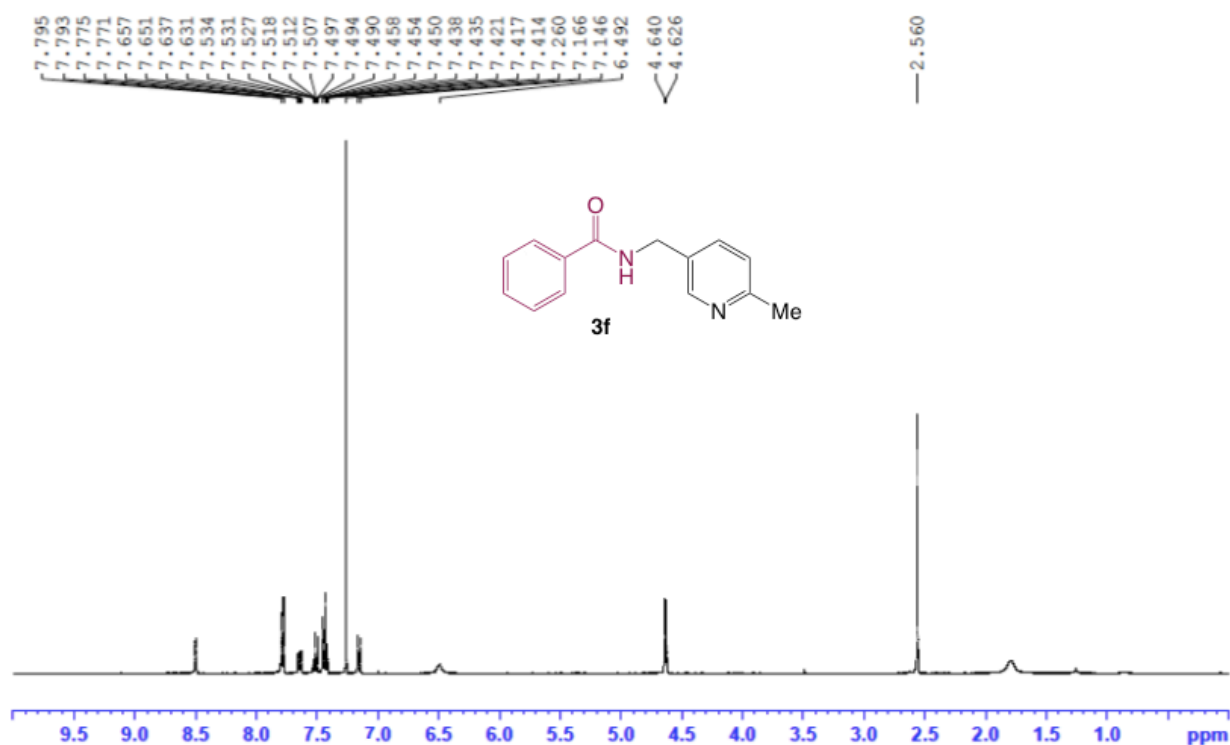


Figure S6b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-((6-methylpyridin-3-yl)methyl)benzamide (**3f**)

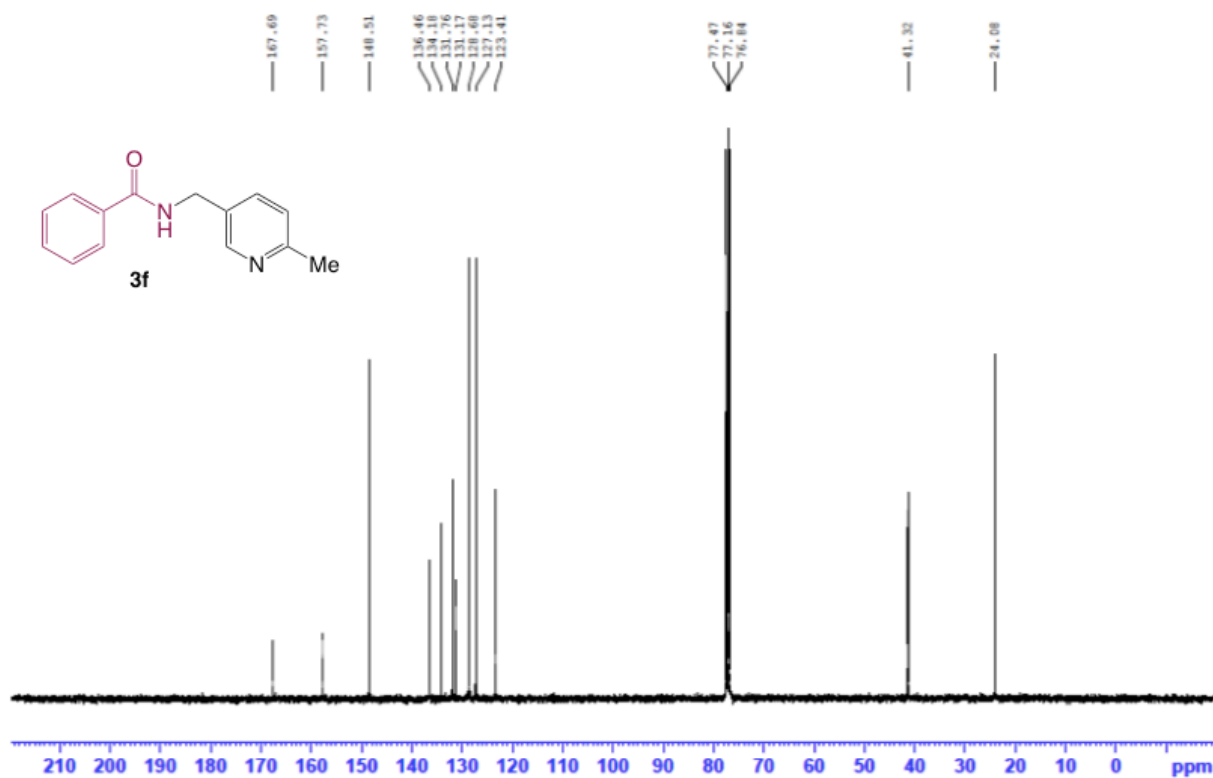


Figure S7a: ^1H NMR (400 MHz, CDCl_3) of *N*-(thiophen-3-ylmethyl)benzamide (**3g**)

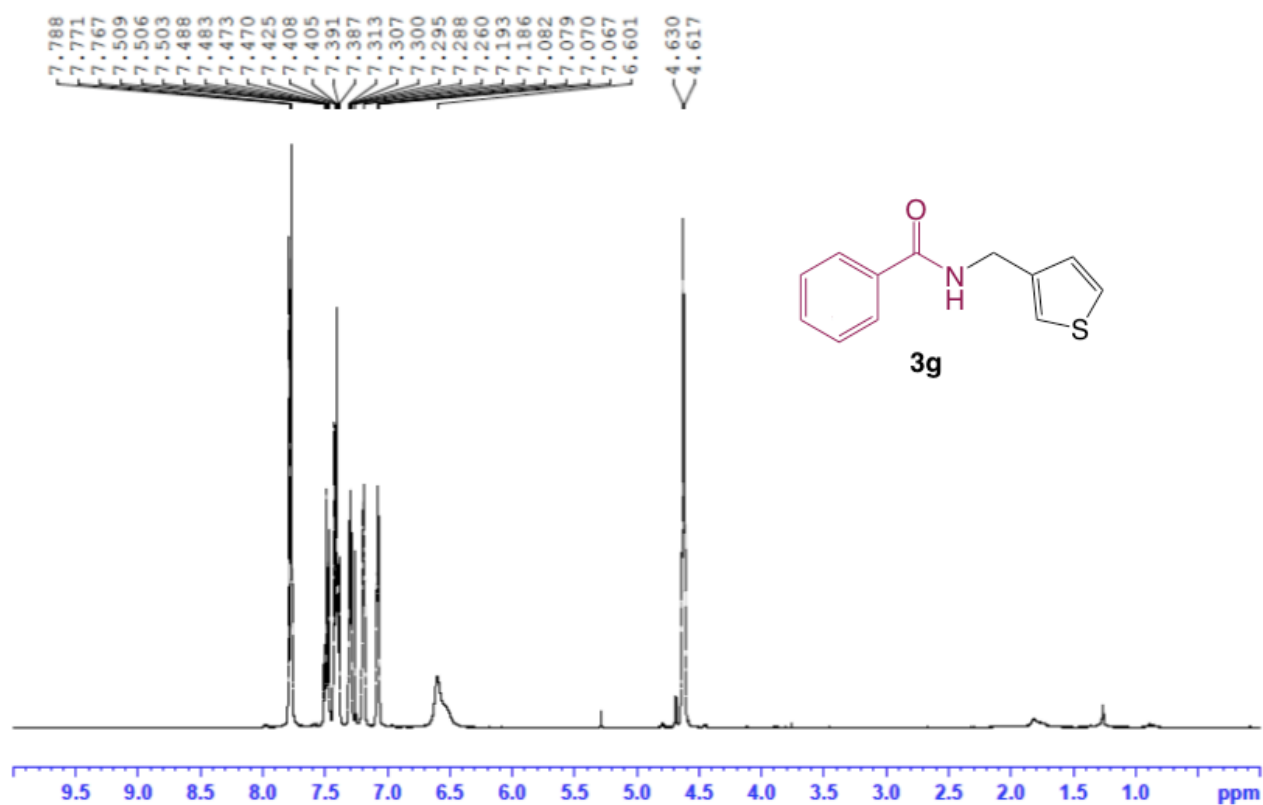


Figure S7b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-(thiophen-3-ylmethyl)benzamide (**3g**)

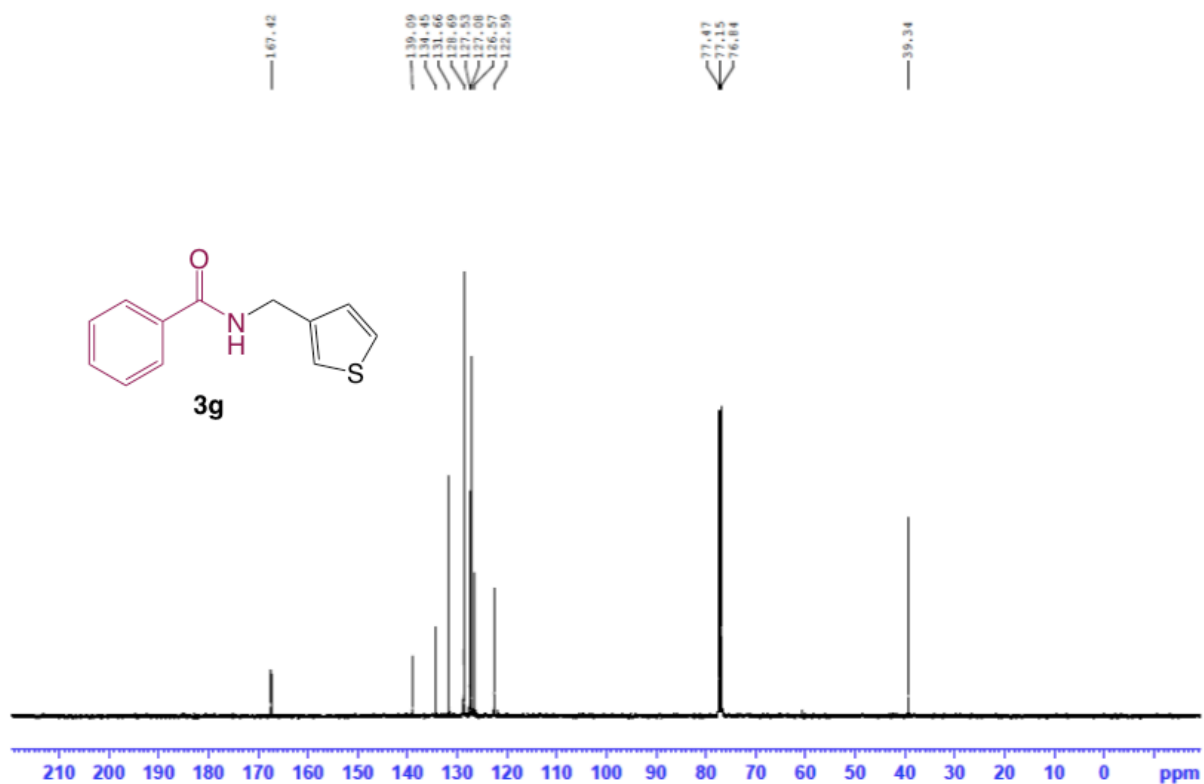


Figure S8a: ^1H NMR (400 MHz, CDCl_3) of *N*-(furan-3-ylmethyl)benzamide (**3h**)

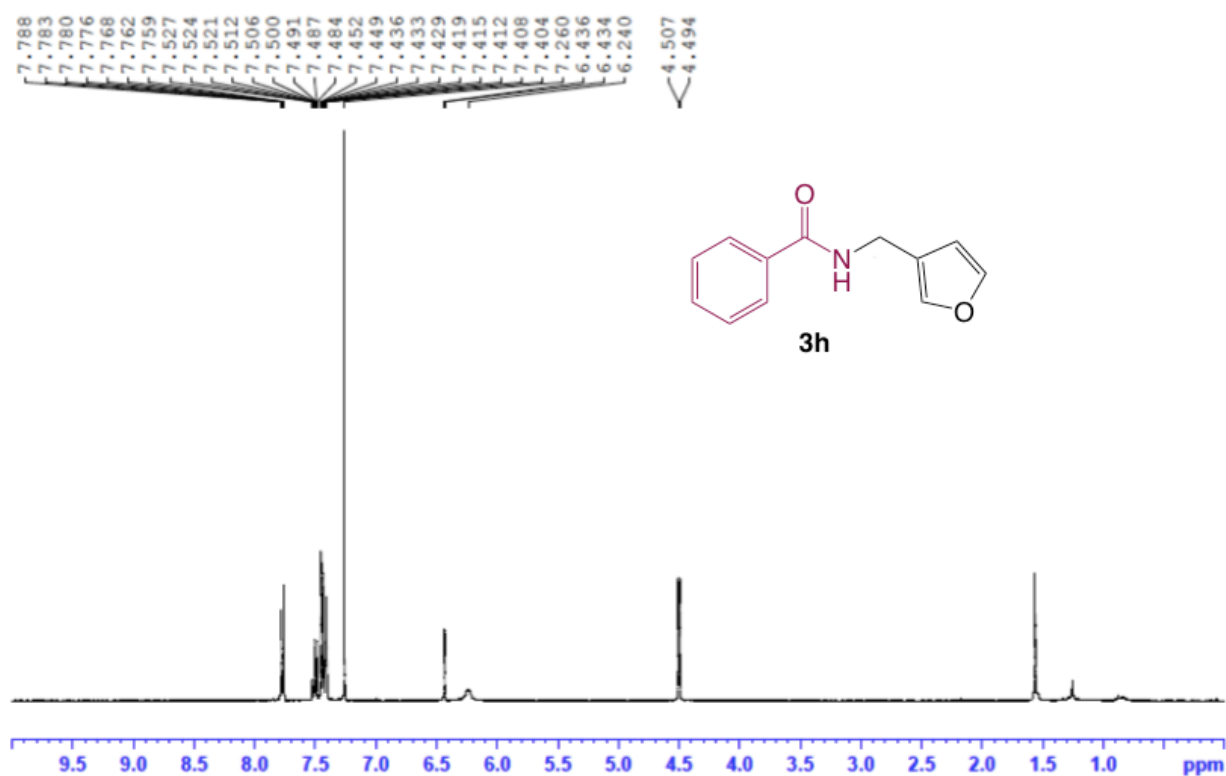


Figure S8b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-(furan-3-ylmethyl)benzamide (**3h**)

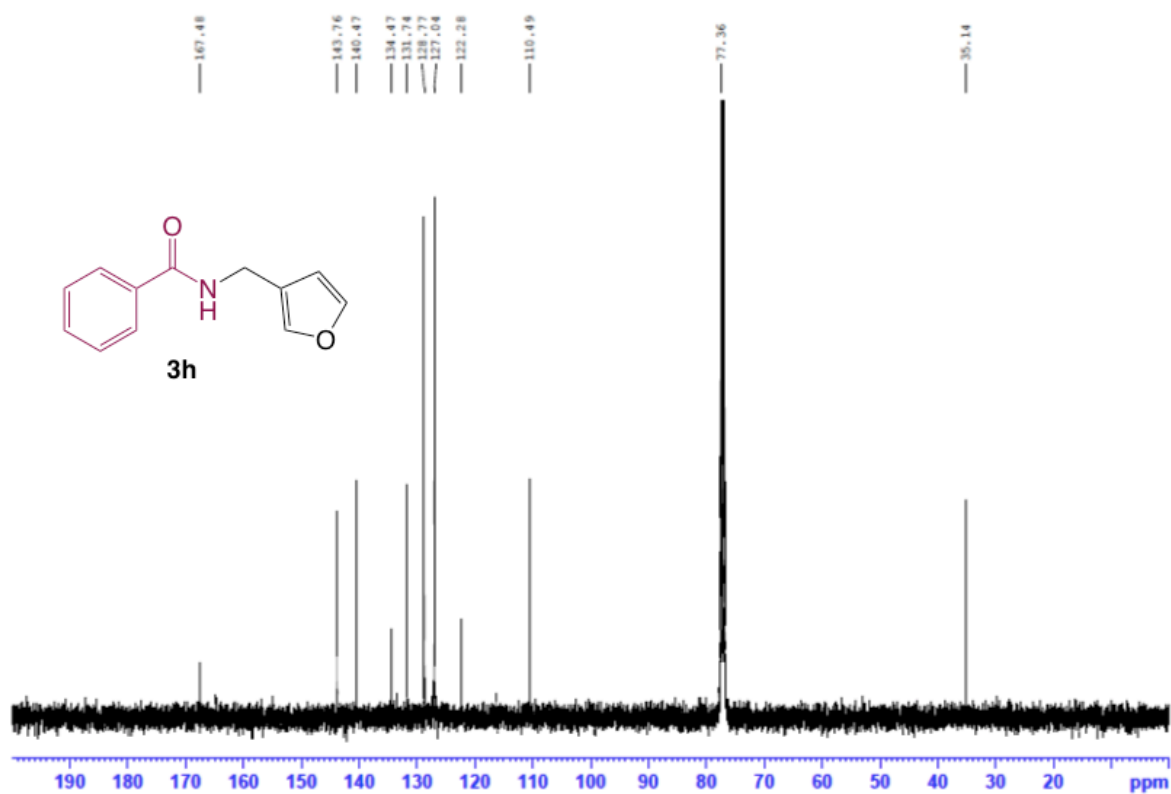


Figure S9a: ^1H NMR (400 MHz, CDCl_3) of 4-methyl-*N*-(3-phenylpropyl)benzamide (**3i**)

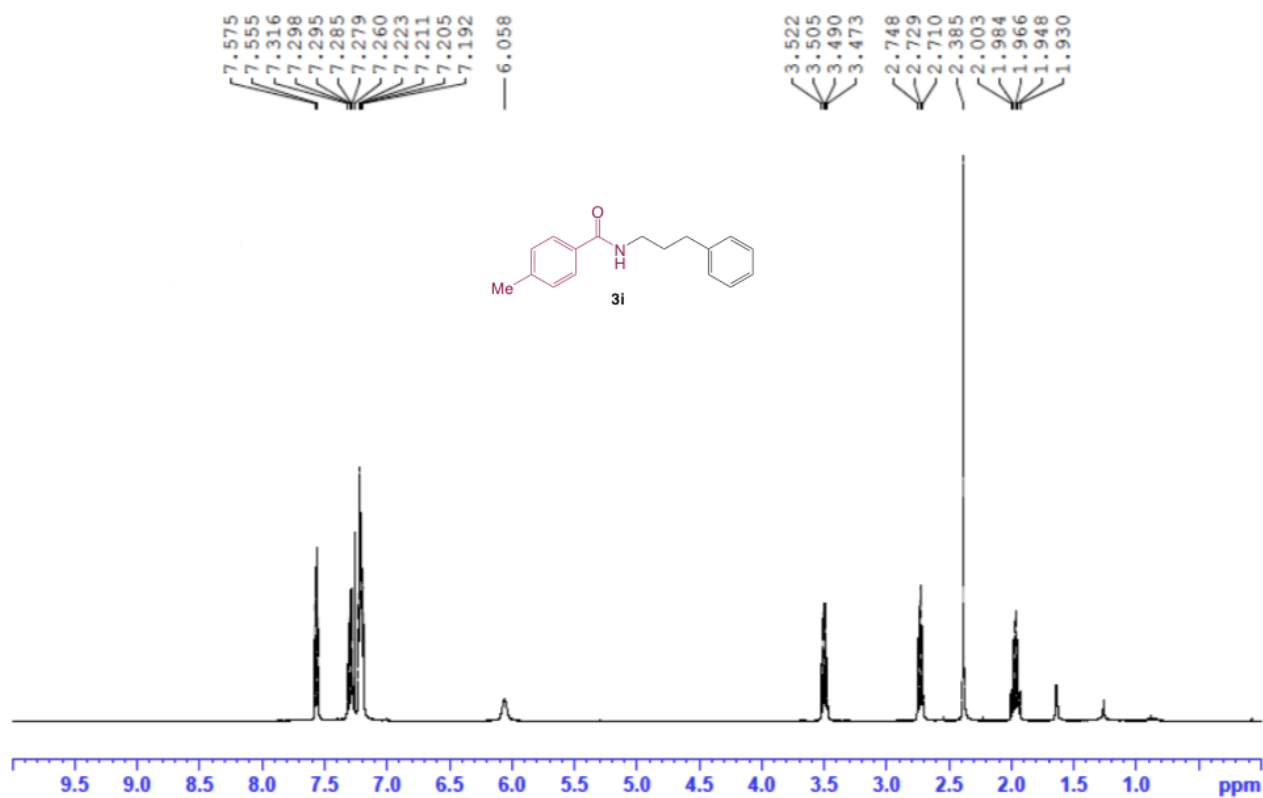


Figure S9b: ^{13}C NMR (100 MHz, CDCl_3) of 4-methyl-*N*-(3-phenylpropyl)benzamide (**3i**)

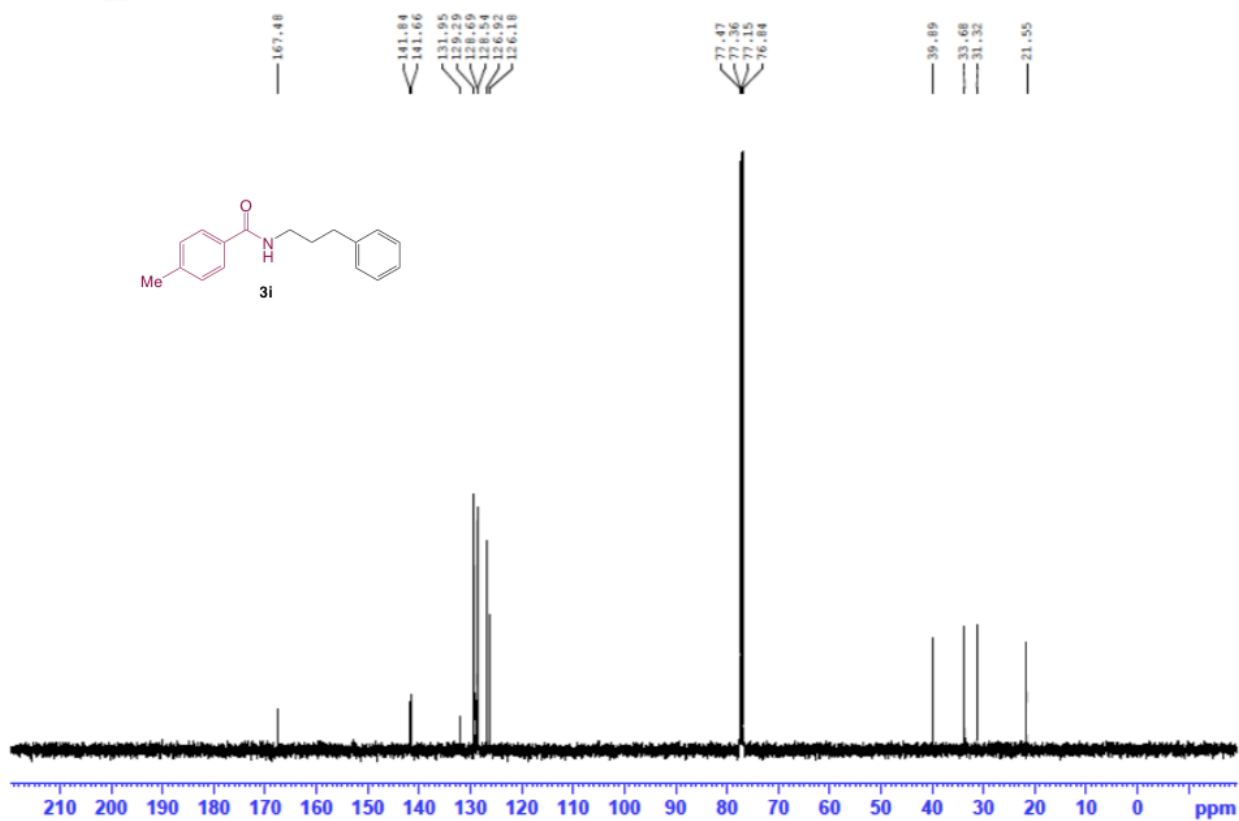


Figure S10a: ^1H NMR (400 MHz, CDCl_3) of *N*-(4-fluorobenzyl)benzamide (**3j**)

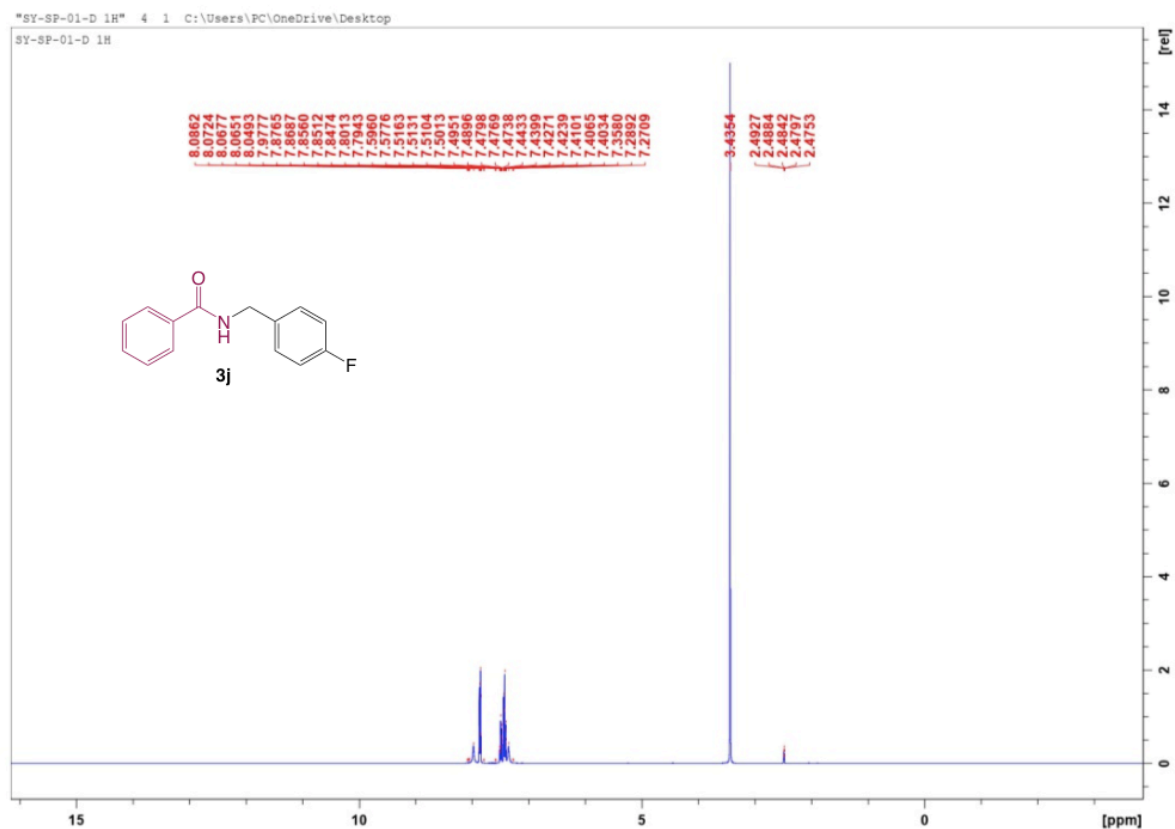


Figure S10b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-(4-fluorobenzyl)benzamide (**3j**)

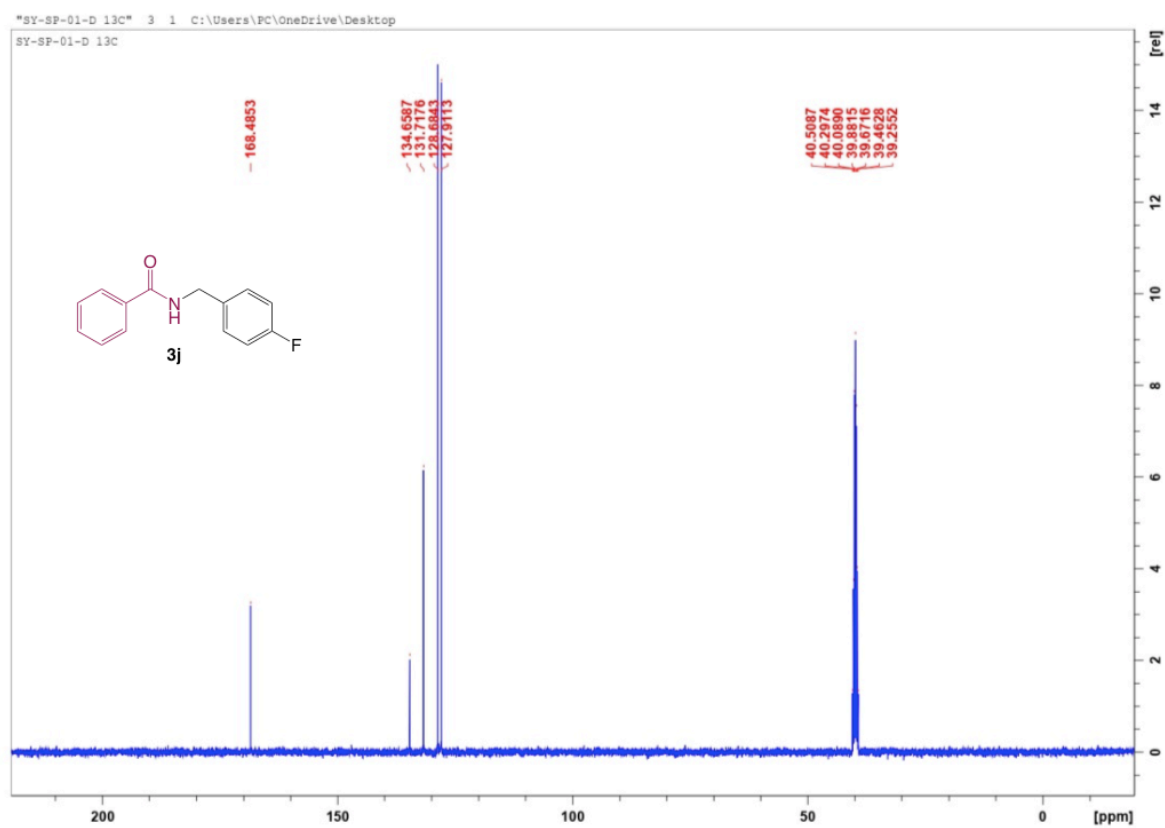


Figure S11a: ^1H NMR (400 MHz, CDCl_3) of *N*-benzyl-4-methoxybenzamide (**3k**)



Figure S11b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-benzyl-4-methoxybenzamide (**3k**)

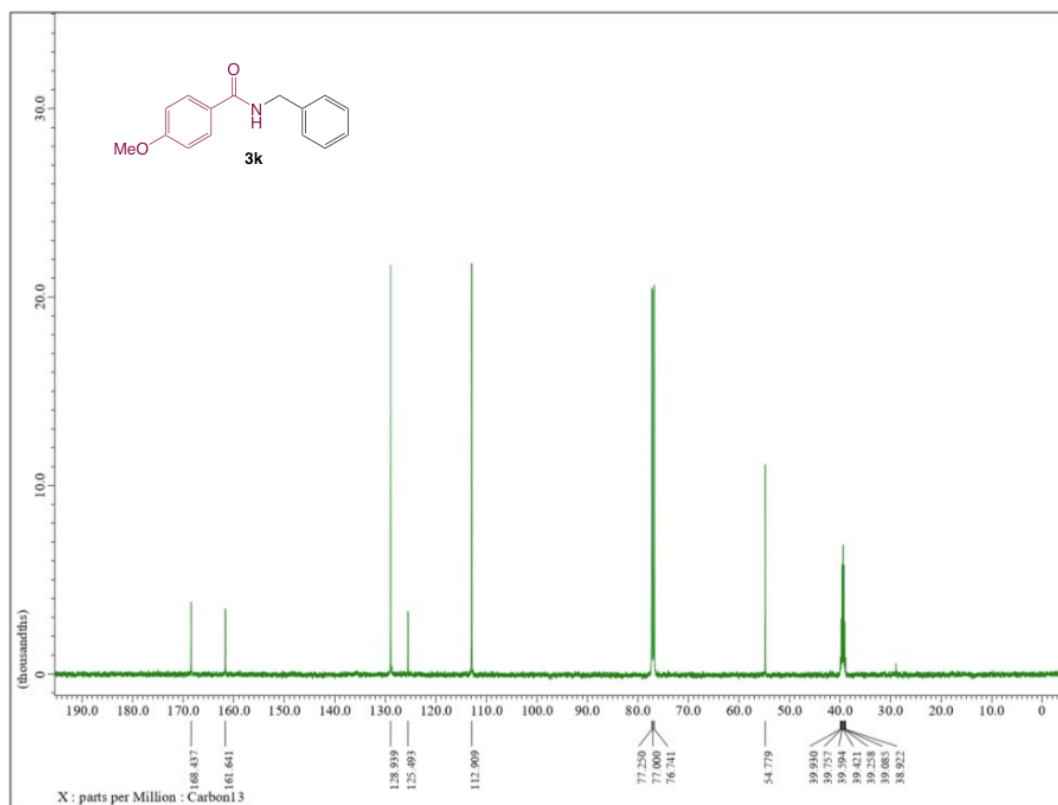


Figure S12a: ^1H NMR (400 MHz, CDCl_3) of *N*-(naphthalen-2-ylmethyl)benzamide (**3I**)

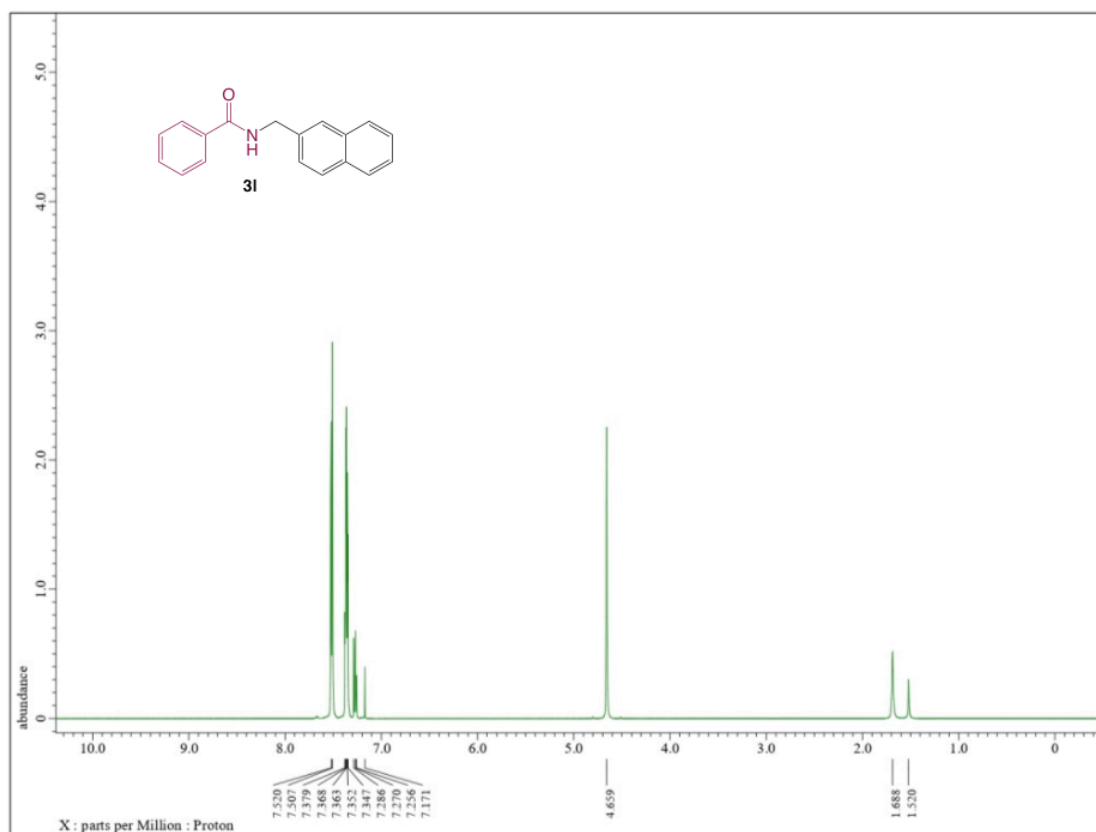


Figure S12b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-(naphthalen-2-ylmethyl)benzamide (**3I**)

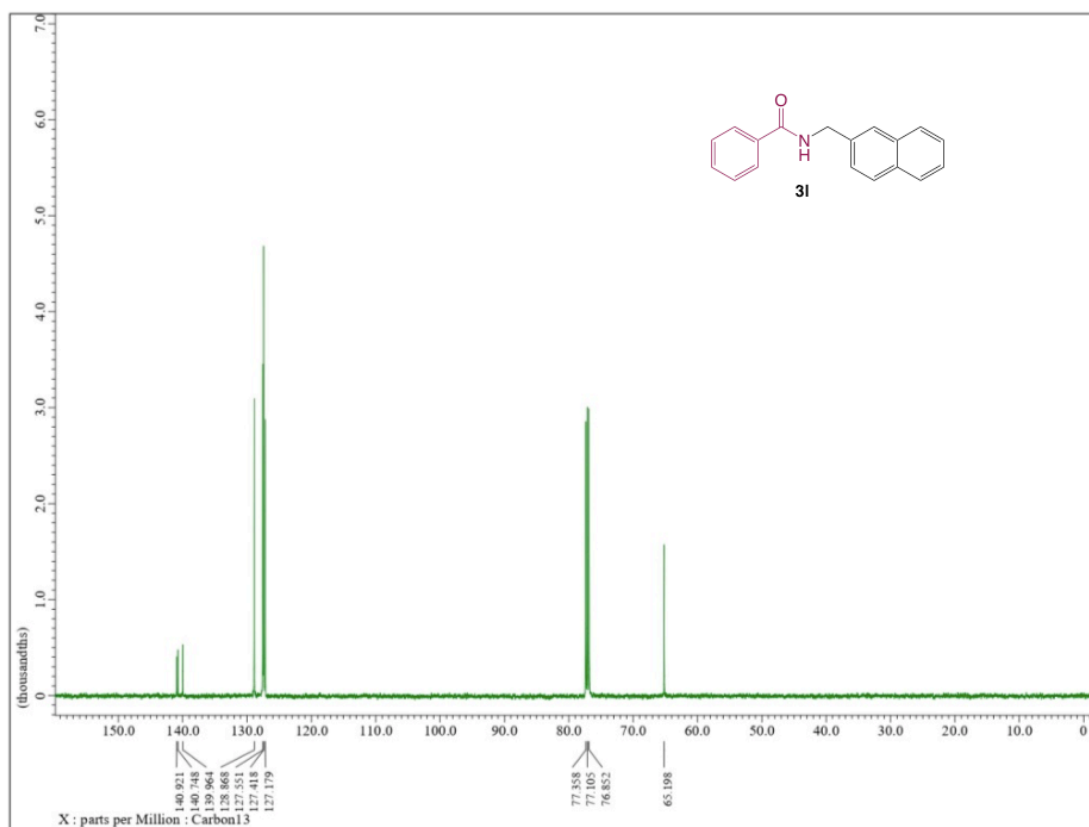


Figure S13a: ^1H NMR (400 MHz, CDCl_3) of *N*-butylhexanamide (**3m**)

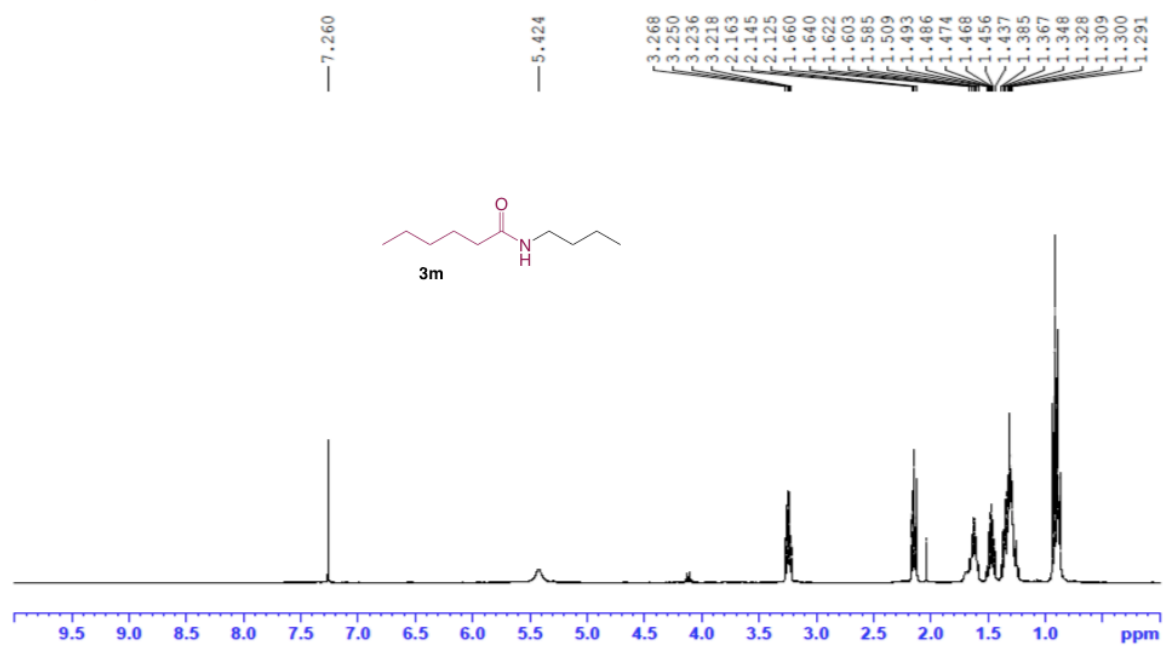


Figure S13b: ^{13}C NMR (100 MHz, CDCl_3) of *N*-butylhexanamide (**3m**)

