

Electronic Supplementary Information (ESI)

An Efficient, One-Pot Transamidation of 8-Aminoquinoline Amides Activated by Boc

Wengang Wu ¹, Jun Yi ^{2,*}, Huipeng Xu ², Shuangjun Li ² and Rongxin Yuan ^{2,*}

¹ Soochow University, 199 Ren'ai Road, Suzhou, Jiangsu 215123, China

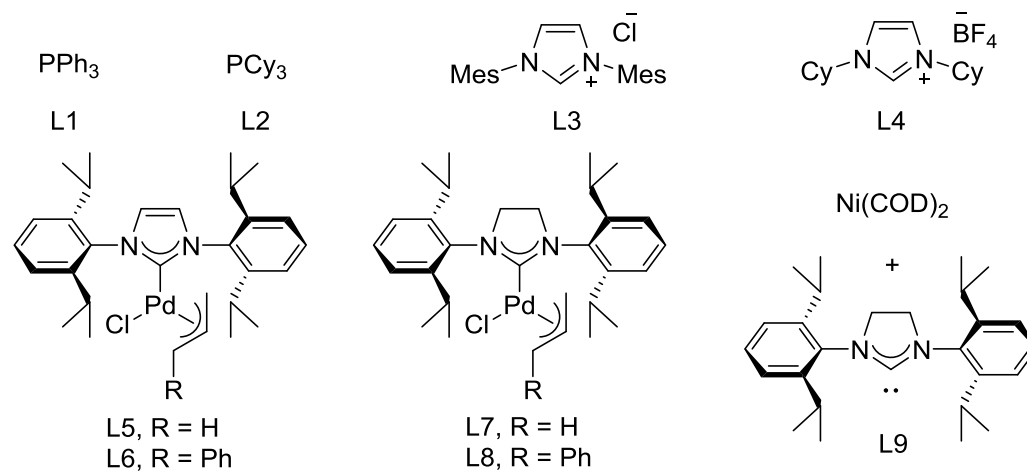
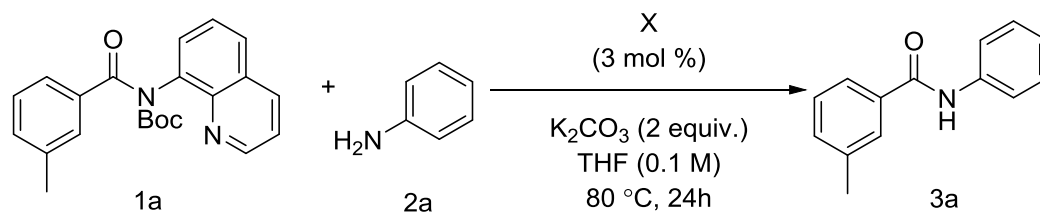
E-mail: 20155209006@suda.edu.cn (W. W.).

² Jiangsu Laboratory of Advanced Functional Material, School of Chemistry and Materials Engineering, Changshu Institute of Technology, Changshu 215500, China

General Methods

Reagent-grade solvents were used for extraction, recrystallization, and flash chromatography. All other commercial reagents were used as received without additional purification. The progress of reactions was checked by analytical thin-layer chromatography (TLC, silica gel 60 F-254 plates) (Qingdao Haiyang Chemical Co., Ltd., Qingdao, Shandong, China). The plates were visualized first with UV illumination, followed by iodine or phosphomolybdic acid hydrate. Column chromatography was performed using silica gel (200–300 mesh) (Qingdao Haiyang Chemical Co., Ltd., Qingdao, Shandong, China). NMR spectra were obtained using a BRUKER AVANCE III instrument (Bruker Co., Ltd., Switzerland). ¹H-NMR spectra were recorded at 400 MHz and are reported in parts per million (ppm) on the δ scale relative to tetramethylsilane (TMS) as an internal standard. ¹³C-NMR spectra were recorded at 100 MHz and are reported in parts per million (ppm) on the δ scale relative to CDCl₃ (δ 77.16). Multiplicities are indicated as the following: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; dd, doubled doublet; td, tripled doublet; br, broad. Coupling constants (J values), where noted, are quoted in hertz. Mass spectra were obtained using an Agilent 1260-6120 (ESI) instrument (Agilent Technologies Co., Ltd., Santa Clara, CA, USA).

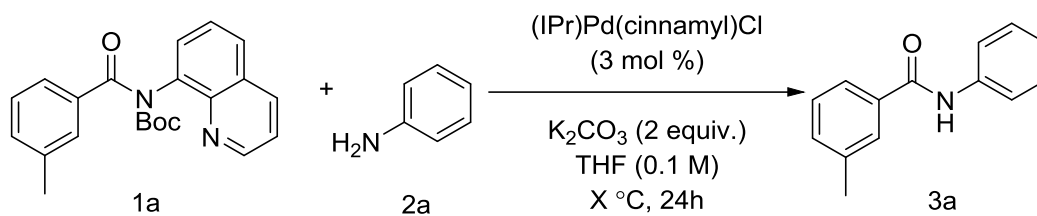
Supplementary Table 1. Screening of catalyst^a.



entry	X	yield of 3 (%) ^a
1	L1+Pd(OAc) ₂	0
2	L2+Pd(OAc) ₂	0
3	L3+Pd(OAc) ₂	14
4	L4+Pd(OAc) ₂	8
5	L5	30
6	L6	44
7	L7	26
8	L8	25
9	L9+Ni(COD) ₂	68

^aReaction conditions (unless otherwise specified): 1a (0.2 mmol, 1.0 equiv), 2a (0.3 mmol, 1.5 equiv), and THF (2 mL). ^bDetermined by mesitylene as an internal standard.

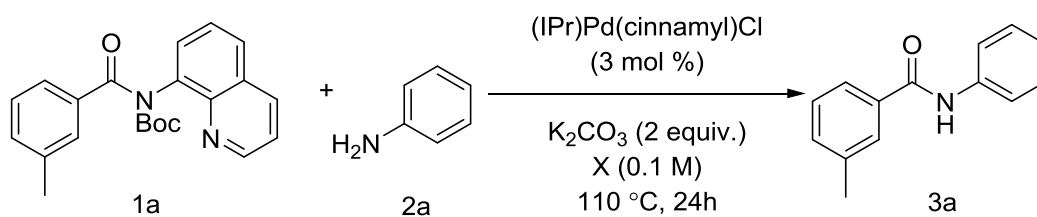
Supplementary Table 2. Screening of reaction temperature^a.



entry	X	yield of 3 (%) ^a
1	40	8
2	80	44
3	110	96
4	130	97

^aReaction conditions (unless otherwise specified): 1a (0.2 mmol, 1.0 equiv), 2a (0.3 mmol, 1.5 equiv), and THF (2 mL). ^bDetermined by mesitylene as an internal standard.

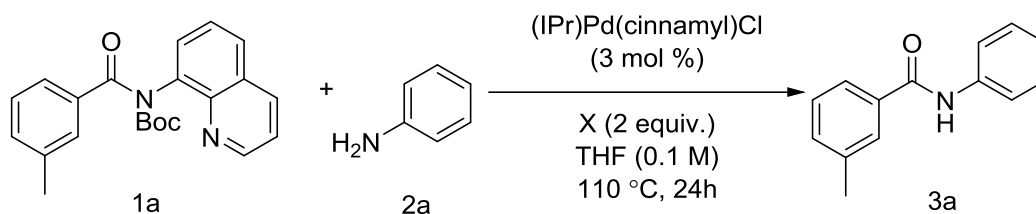
Supplementary Table 3. Screening of solvent^a.



entry	X	yield of 3 (%) ^a
1	CH ₂ Cl ₂	27
2	THF	96
3	DMF	72
4	DMAc	80
5	1, 4 - Dioxane	85
6	CH ₃ CN	70

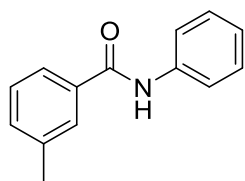
^aReaction conditions (unless otherwise specified): 1a (0.2 mmol, 1.0 equiv), 2a (0.3 mmol, 1.5 equiv), and solvent (2 mL). ^bDetermined by mesitylene as an internal standard.

Supplementary Table 4. Screening of base^a.



entry	X	yield of 3 (%) ^a
1	K ₃ PO ₄	27
2	K ₂ CO ₃	96
3	NaOH	72
4	<i>t</i> BuONa	80
5	Cs ₂ CO ₃	85
6	DBU	70

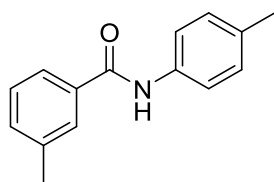
^aReaction conditions (unless otherwise specified): 1a (0.2 mmol, 1.0 equiv), 2a (0.3 mmol, 1.5 equiv), and solvent (2 mL). ^bDetermined by mesitylene as an internal standard.



3-methyl-N-phenylbenzamide (3a):

¹H NMR (400 MHz, Chloroform-*d*) δ 7.90 (s, 1H), 7.70 – 7.59 (m, 4H), 7.40 – 7.30 (m, 4H), 7.14 (t, *J* = 7.4 Hz, 1H), 2.41 (s, 3H).

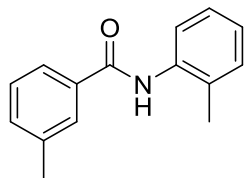
¹³C NMR (101 MHz, CDCl₃) δ 166.0, 138.7, 138.0, 135.0, 132.5, 129.0, 128.6, 127.8, 124.5, 123.9, 120.2, 21.4.



3-methyl-N-(p-tolyl)benzamide (3b):

¹H NMR (400 MHz, Chloroform-*d*) δ 7.77 (s, 1H), 7.68 (s, 1H), 7.63 (dt, *J* = 5.7, 2.2 Hz, 1H), 7.58 – 7.50 (m, 2H), 7.39 – 7.31 (dd, *J* = 5.0, 1.9 Hz, 2H), 7.17 (d, *J* = 8.1 Hz, 2H), 2.42 (s, 3H), 2.34 (s, 3H).

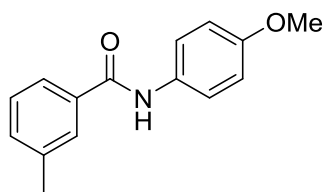
¹³C NMR (101 MHz, CDCl₃) δ 165.8, 138.6, 135.4, 135.1, 134.1, 132.4, 129.5, 128.6, 127.7, 123.9, 120.2, 21.4, 20.9.



3-methyl-*N*-(*o*-tolyl)benzamide (3c):

^1H NMR (400 MHz, Chloroform-*d*) δ 7.93 (d, J = 8.0 Hz, 1H), 7.76 – 7.59 (m, 3H), 7.37 (d, J = 5.5 Hz, 2H), 7.29 – 7.19 (m, 2H), 7.11 (t, J = 7.5 Hz, 1H), 2.44 (s, 3H), 2.33 (s, 3H).

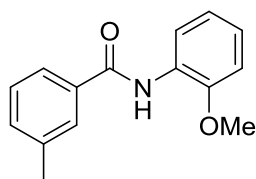
^{13}C NMR (101 MHz, CDCl_3) δ 165.8, 138.8, 135.8, 135.0, 132.6, 130.5, 129.2, 128.6, 127.9, 126.9, 125.3, 123.9, 123.1, 21.4, 17.8.



N-(4-methoxyphenyl)-3-methylbenzamide (3d):

^1H NMR (400 MHz, Chloroform-*d*) δ 7.69 (d, J = 8.8 Hz, 2H), 7.63 (d, J = 6.3 Hz, 1H), 7.58 – 7.50 (m, 2H), 7.40 – 7.31 (m, 2H), 6.95 – 6.86 (m, 2H), 3.82 (s, 3H), 2.43 (s, 3H).

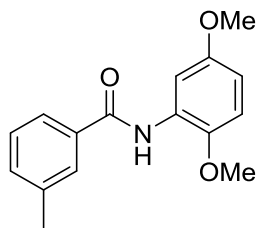
^{13}C NMR (101 MHz, CDCl_3) δ 166.0, 156.5, 138.5, 134.9, 132.3, 131.1, 128.4, 127.8, 124.0, 122.2, 114.1, 55.4, 21.3.



N-(2-methoxyphenyl)-3-methylbenzamide (3e):

^1H NMR (400 MHz, Chloroform-*d*) δ 8.53 (d, J = 6.6 Hz, 2H), 7.75 – 7.69 (m, 1H), 7.66 (d, J = 6.8 Hz, 1H), 7.37 (d, J = 7.3 Hz, 2H), 7.12 – 6.96 (m, 2H), 6.92 (d, J = 7.9 Hz, 1H), 3.93 (s, 3H), 2.44 (s, 3H).

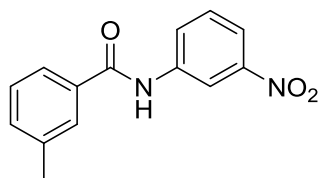
^{13}C NMR (101 MHz, CDCl_3) δ 165.5, 148.1, 138.6, 135.3, 132.4, 128.6, 127.8, 127.8, 123.9, 123.8, 121.2, 119.8, 109.9, 55.8, 21.4.



N-(2,5-dimethoxyphenyl)-3-methylbenzamide (3f):

^1H NMR (400 MHz, Chloroform-*d*) δ 8.56 (s, 1H), 8.29 (d, $J = 3.0$ Hz, 1H), 7.78 – 7.62 (m, 2H), 7.37 (d, $J = 7.0$ Hz, 2H), 6.83 (d, $J = 8.9$ Hz, 1H), 6.61 (dd, $J = 8.9, 3.0$ Hz, 1H), 3.88 (s, 3H), 3.82 (s, 3H), 2.44 (s, 3H).

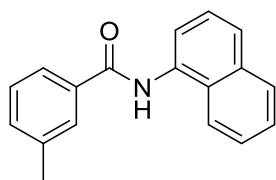
^{13}C NMR (101 MHz, CDCl_3) δ 165.4, 153.9, 142.3, 138.7, 135.2, 132.5, 128.6, 128.5, 127.8, 123.9, 110.7, 108.8, 105.9, 56.3, 55.8, 21.4.



3-methyl-*N*-(3-nitrophenyl)benzamide (3g):

^1H NMR (400 MHz, Chloroform-*d*) δ 8.49 (t, $J = 2.1$ Hz, 1H), 8.22 (s, 1H), 8.11 (d, $J = 8.2$ Hz, 1H), 7.98 (d, $J = 8.2$ Hz, 1H), 7.74 – 7.62 (m, 2H), 7.52 (t, $J = 8.2$ Hz, 1H), 7.37 (d, $J = 5.3$ Hz, 2H), 2.42 (s, 3H).

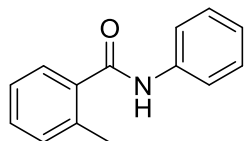
^{13}C NMR (101 MHz, CDCl_3) δ 166.2, 148.6, 139.1, 138.9, 133.9, 133.2, 129.9, 128.8, 127.8, 125.9, 124.1, 119.0, 114.9, 21.3.



3-methyl-*N*-(naphthalen-1-yl)benzamide (3h):

^1H NMR (400 MHz, Chloroform-*d*) δ 8.20 (s, 1H), 8.03 (d, $J = 7.5$ Hz, 1H), 7.90 (dt, $J = 6.5, 2.9$ Hz, 2H), 7.84 – 7.70 (m, 3H), 7.52 (td, $J = 8.0, 7.3, 4.4$ Hz, 3H), 7.41 (d, $J = 6.0$ Hz, 2H), 2.46 (s, 3H).

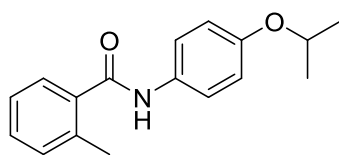
^{13}C NMR (101 MHz, CDCl_3) δ 166.5, 138.6, 134.7, 134.1, 132.6, 132.4, 128.7, 128.6, 128.0, 127.6, 126.2, 126.0, 125.9, 125.7, 124.1, 121.4, 120.9, 21.3.



2-methyl-*N*-phenylbenzamide (3i):

^1H NMR (400 MHz, Chloroform-*d*) δ 7.65 (d, J = 7.9 Hz, 2H), 7.57 (s, 1H), 7.50 (d, J = 7.6 Hz, 1H), 7.39 (t, J = 8.0 Hz, 3H), 7.29 (t, J = 5.3 Hz, 2H), 7.18 (t, J = 7.5 Hz, 1H), 2.53 (s, 3H).

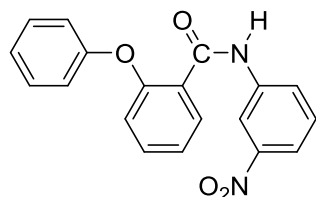
^{13}C NMR (101 MHz, CDCl_3) δ 168.2, 138.0, 136.4, 131.2, 130.2, 129.1, 126.6, 125.9, 124.5, 119.9, 19.8.



N-(4-isopropoxyphenyl)-2-methylbenzamide (3j):

^1H NMR (400 MHz, Chloroform-*d*) δ 7.84 – 7.61 (m, 3H), 7.54 (d, J = 8.4 Hz, 2H), 7.44 – 7.32 (m, 2H), 6.92 (d, J = 8.4 Hz, 2H), 4.63 – 4.47 (m, 1H), 2.45 (s, 3H), 1.36 (d, J = 6.0 Hz, 6H).

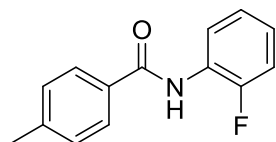
^{13}C NMR (101 MHz, CDCl_3) δ 166.2, 154.7, 138.4, 134.9, 132.3, 131.1, 128.4, 127.9, 124.1, 122.3, 116.3, 70.2, 22.0, 21.3.



N-(3-nitrophenyl)-2-phenoxybenzamide (3k):

^1H NMR (400 MHz, Chloroform-*d*) δ 9.90 (s, 1H), 8.50 (d, J = 2.8 Hz, 1H), 8.35 (d, J = 7.9 Hz, 1H), 7.98 (dd, J = 14.1, 8.4 Hz, 2H), 7.47 (td, J = 10.7, 9.1, 5.7 Hz, 4H), 7.29 (d, J = 7.5 Hz, 2H), 7.16 (d, J = 8.0 Hz, 2H), 6.90 (d, J = 8.3 Hz, 1H).

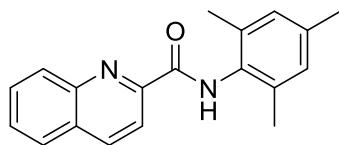
^{13}C NMR (101 MHz, CDCl_3) δ 163.1, 155.6, 154.9, 148.6, 139.2, 133.7, 132.6, 130.5, 129.7, 126.0, 125.4, 124.0, 123.0, 119.8, 118.9, 118.2, 115.1.



N-(2-fluorophenyl)-4-methylbenzamide (3l):

^1H NMR (400 MHz, Chloroform-*d*) δ 8.40 (d, $J = 15.6$ Hz, 1H), 8.24 – 8.12 (m, 1H), 7.60 – 7.46 (m, 3H), 7.31 (t, $J = 7.6$ Hz, 1H), 7.23 – 7.12 (m, 3H), 2.35 (s, 3H).

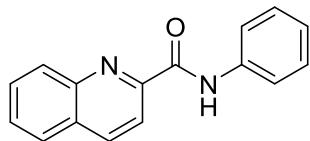
^{13}C NMR (101 MHz, Chloroform-*d*) δ 161.2, 160.4 (d, $J = 246.2$ Hz), 135.1, 134.4, 133.6 (d, $J = 9.4$ Hz), 132.3 (d, $J = 2.0$ Hz), 129.5, 125.0 (d, $J = 3.3$ Hz), 121.4 (d, $J = 11.1$ Hz), 120.5, 116.1 (d, $J = 25.1$ Hz).



N-mesitylquinoline-2-carboxamide (3m):

^1H NMR (400 MHz, Chloroform-*d*) δ 9.67 (s, 1H), 8.41 (q, $J = 8.6$ Hz, 2H), 8.20 (d, $J = 8.5$ Hz, 1H), 7.95 (d, $J = 8.2$ Hz, 1H), 7.83 (t, $J = 7.8$ Hz, 1H), 7.68 (t, $J = 7.6$ Hz, 1H), 6.99 (s, 2H), 2.34 (s, 3H), 2.32 (s, 6H).

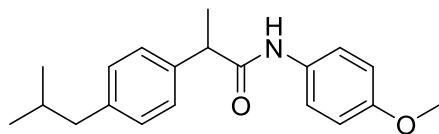
^{13}C NMR (101 MHz, CDCl_3) δ 162.7, 149.7, 146.5, 137.6, 136.8, 135.2, 131.2, 130.2, 129.8, 129.4, 128.9, 128.0, 127.8, 119.0, 21.0, 18.6.



N-phenylquinoline-3-carboxamide (3n):

^1H NMR (400 MHz, Chloroform-*d*) δ 10.27 (s, 1H), 8.41 (q, $J = 8.5$ Hz, 2H), 8.22 (d, $J = 8.5$ Hz, 1H), 8.00 – 7.78 (m, 4H), 7.68 (t, $J = 7.6$ Hz, 1H), 7.45 (t, $J = 7.8$ Hz, 2H), 7.21 (t, $J = 7.4$ Hz, 1H).

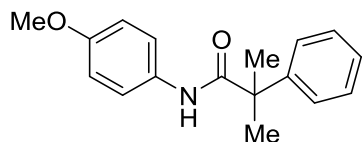
^{13}C NMR (101 MHz, CDCl_3) δ 162.1, 149.6, 146.3, 137.9, 137.8, 130.3, 129.7, 129.4, 129.1, 128.2, 127.8, 124.3, 119.7, 118.7.



2-(4-isobutylphenyl)-*N*-(4-methoxyphenyl)propanamide (3o):

^1H NMR (400 MHz, Chloroform-*d*) δ 7.31 (d, $J = 8.6$ Hz, 2H), 7.26 (d, $J = 7.2$ Hz, 2H), 7.15 (d, $J = 7.7$ Hz, 2H), 6.97 (s, 1H), 6.80 (d, $J = 8.6$ Hz, 2H), 3.76 (s, 3H), 3.67 (q, $J = 7.2$ Hz, 1H), 2.47 (d, $J = 7.1$ Hz, 2H), 1.95 – 1.79 (m, 1H), 1.58 (d, $J = 7.2$ Hz, 3H), 0.91 (d, $J = 6.6$ Hz, 6H).

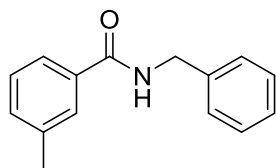
^{13}C NMR (101 MHz, CDCl_3) δ 172.5, 156.3, 140.9, 138.2, 131.1, 129.7, 127.4, 121.6, 114.0, 55.4, 47.4, 45.0, 30.1, 22.3, 18.5.



N-(4-methoxyphenyl)-2-methyl-2-phenylpropanamide (3p):

^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 9.00 (s, 1H), 7.47 (d, $J = 8.5$ Hz, 2H), 7.42 – 7.30 (m, 4H), 7.24 (dt, $J = 6.4, 3.1$ Hz, 1H), 6.84 (d, $J = 8.8$ Hz, 2H), 3.70 (s, 3H), 1.55 (s, 6H).

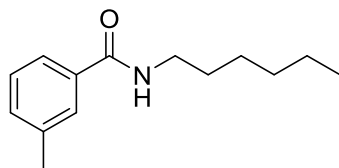
^{13}C NMR (101 MHz, DMSO) δ 174.7, 155.7, 146.5, 132.7, 128.7, 126.7, 126.1, 122.3, 113.9, 55.5, 47.5, 27.3.



N-benzyl-3-methylbenzamide (3q):

^1H NMR (400 MHz, $\text{Chloroform}-d$) δ 7.61 (s, 1H), 7.57 (s, 1H), 7.39 – 7.26 (m, 7H), 6.56 (s, 1H), 4.61 (d, $J = 5.7$ Hz, 2H), 2.37 (s, 3H).

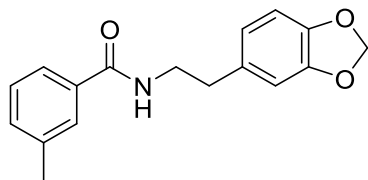
^{13}C NMR (101 MHz, CDCl_3) δ 167.6, 138.4, 138.3, 134.3, 132.2, 128.7, 128.4, 127.9, 127.7, 127.5, 123.9, 44.1, 21.3.



N-hexyl-3-methylbenzamide (3r):

^1H NMR (400 MHz, $\text{Chloroform}-d$) δ 7.51 (s, 1H), 7.49 – 7.42 (m, 1H), 7.20 (d, $J = 4.7$ Hz, 2H), 6.30 (s, 1H), 3.44 – 3.27 (m, 2H), 2.29 (s, 3H), 1.58 – 1.44 (m, 2H), 1.32 – 1.16 (m, 6H), 0.87 – 0.75 (m, 3H).

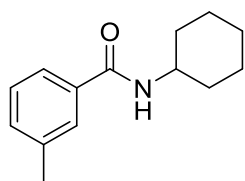
^{13}C NMR (101 MHz, CDCl_3) δ 167.8, 138.2, 134.8, 131.9, 128.3, 127.7, 123.8, 40.1, 31.5, 29.6, 26.6, 22.5, 21.3, 14.0.



N-(benzo[d][1,3]dioxol-5-yl)-3-methylbenzamide (3s):

^1H NMR (400 MHz, Chloroform-*d*) δ 7.54 (s, 1H), 7.49 – 7.42 (m, 1H), 7.28 – 7.24 (m, 2H), 6.81 – 6.60 (m, 3H), 6.26 (s, 1H), 3.64 (q, $J = 6.6$ Hz, 2H), 2.83 (t, $J = 6.9$ Hz, 2H), 2.37 (s, 3H).

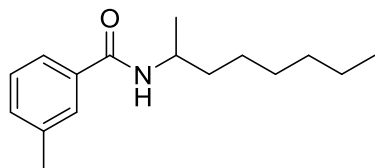
^{13}C NMR (101 MHz, CDCl_3) δ 167.7, 147.9, 146.2, 138.4, 134.6, 132.6, 132.1, 128.4, 127.6, 123.7, 121.7, 109.1, 108.3, 100.9, 41.3, 35.4, 21.3.



N-cyclohexyl-3-methylbenzamide (3t):

^1H NMR (400 MHz, Chloroform-*d*) δ 7.57 (s, 1H), 7.55 – 7.48 (m, 1H), 7.35 – 7.27 (m, 2H), 5.93 (s, 1H), 4.05 – 3.90 (m, 1H), 2.40 (s, 3H), 2.10 – 1.96 (m, 2H), 1.84 – 1.56 (m, 4H), 1.52 – 1.35 (m, 2H), 1.29 – 1.18 (m, 2H).

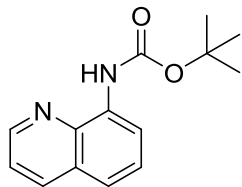
^{13}C NMR (101 MHz, CDCl_3) δ 166.8, 138.2, 135.1, 131.8, 128.3, 127.6, 123.8, 48.6, 33.1, 25.5, 24.9, 21.3.



3-methyl-*N*-(octan-2-yl)benzamide (3u):

^1H NMR (400 MHz, Chloroform-*d*) δ 7.59 (s, 1H), 7.54 (t, $J = 4.6$ Hz, 1H), 7.26 (d, $J = 4.6$ Hz, 2H), 6.53 (s, 1H), 3.36 (td, $J = 6.2, 2.0$ Hz, 2H), 2.35 (s, 3H), 1.65 – 1.48 (q, $J = 6.1$ Hz, 1H), 1.42 – 1.23 (m, 8H), 0.98 – 0.80 (m, 6H).

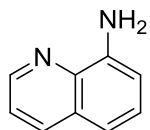
^{13}C NMR (101 MHz, CDCl_3) δ 167.9, 138.2, 134.9, 131.9, 128.3, 127.7, 123.8, 43.0, 39.4, 31.0, 28.9, 24.3, 23.0, 21.3, 14.0, 10.8.



tert-butyl quinolin-8-ylcarbamate (3v):

^1H NMR (400 MHz, Chloroform-*d*) δ 9.05 (s, 1H), 8.82 (dd, $J = 4.1, 2.1$ Hz, 1H), 8.44 (d, $J = 7.6$ Hz, 1H), 8.16 (d, $J = 8.3$ Hz, 1H), 7.54 (t, $J = 8.0$ Hz, 1H), 7.45 (d, $J = 7.8$ Hz, 2H), 1.60 (s, 9H).

^{13}C NMR (101 MHz, CDCl_3) δ 152.94, 148.00, 138.24, 136.30, 135.18, 128.07, 127.38, 121.56, 120.18, 114.42, 80.45, 28.43.



8-aminequinoline (3x):

