Convenient synthesis of amides by $Zn(ClO_4)_2·6H_2O$ catalysed Ritter reaction with nitriles and halohydrocarbons

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A convenient and high yielding procedure for the synthesis of amides by the Ritter reaction of nitriles and halohydrocarbons in the presence of $Zn(ClO_4)_2·6H_2O$ as a highly stable, effective and available catalyst is described.

Keywords: synthesis of amides, Ritter reaction, $Zn(ClO_4)_2·6H_2O$, halohydrocarbons

The Ritter reaction is the reaction of nitriles with carbocations to produce a variety of amides in the presence of stoichiometric amounts of strong acid.[1,2] The large amount of toxic and corrosive acids used in the reaction in order to produce the carbocation limits its application. Over the past two decades, vast progress has been made in the development of catalysts of the Ritter reaction. These catalysts include boron trifluoride,[3] nafion-H,[4] silica sulfuric acid,[5] *o*-benzenedisulfonimide,[6] iodine (I_2),[7] ferric trichloride ($FeCl_3$),[8] cobalt(II) chloride ($CoCl_2$),[9] Fe^{3+}-montmodlonite[10] and 2,4-dinitrobenzenesulfonic acid.[11]

The classical Ritter reaction is the reaction of nitriles with alcohols or alkenes to produce amides. In addition, halohydrocarbons also have been employed in the Ritter reaction, first reported by Olah and co-workers in 1979.[12] Since then the Ritter reaction with halohydrocarbons has been rarely studied until Bi *et al*.[13] reported a photo-Ritter-type reaction with five aryl methyl bromides in 2010. Afterwards, Kalkhambkar *et al*.[14] employed the $[BMIM][PF_6]/NOPF_6$ system for the synthesis of amides by the reaction of nitriles with bromides in 2011. Then, Qu *et al*.[15] reported a Lewis acid system for the reaction of nitriles with halohydrocarbons in 2012. These studies effectively expand the scope of the substrates and catalysts of the Ritter reaction. Halohydrocarbons are widely applied in organic synthesis and drug synthesis. Although these catalytic systems are effective, the catalysts for this transformation are very few, which is especially true of Lewis acid catalysts. There is a need to find new catalysts of this type for the reaction of nitriles with halohydrocarbons.

Zinc(II) perchlorate hexahydrate [$Zn(ClO_4)_2·6H_2O$] is a new and highly efficient Lewis acid catalyst that has been used to catalyse some organic reactions, *e.g.* direct β-amination of β-dicarbonyl compounds using iodosobenzene and *p*-toluenesulfonamide,[16] opening of epoxide rings by amines to synthesise (*RS*)/(*R*)-propranolols and (*RS*)/(*R*)/(*S*)-naftopidils,[17] one-pot amination–annulation of α-cyanomethyl-β-ketoesters,[18] conjugate addition of thiols to α,β-unsaturated ketones, condensation of carboxylic acids with alcohols,[19] conversion of β-ketoesters into β-enamino esters,[20] acylation of alcohols with acid anhydrides[21] and Friedel–Crafts reaction of indoles with imines.[22] In addition, $Zn(ClO_4)_2·6H_2O$ is stable to air moisture and can be handled under wet conditions without any loss of efficiency (see SAFETY CAUTION in Experimental).[21]

We report here an efficient procedure for the transformation of nitriles with halohydrocarbons (benzyl, *tert*-butyl and secondary alkyl halohydrocarbons) employing $Zn(ClO_4)_2·6H_2O$ as an economically effective catalyst.

Results and discussion

In our initial study, the reaction of benzyl chloride and benzonitrile was examined using $Zn(OTf)_2$ at 100 °C under solvent-free conditions. It was found that the reaction produced a pale yellow mixture after 5 h and the expected product *N*-benzylbenzamide (**1a**) was obtained in only 53% yield (Table 1, entry 1). To improve the efficiency of the reaction, several Zn Lewis acids were tried which were believed to be able to catalyse this reaction. $ZnCl_2$, a commonly used Lewis acid, was first tried and it afforded **1a** in a slightly improved yield of 67% (Table 1, entry 2). The use of $ZnBr_2$ greatly facilitated the reaction, which gave **1a** in 78% yield after 5 h (Table 1, entry 3). The employment of $Zn(ClO_4)_2·6H_2O$ led to a cleaner reaction and produced **1a** with the highest yield of 92% after 5 h (Table 1, entry 4). On the other hand, some other Zn catalysts like $ZnSO_4$, ZnI_2, $Zn(acac)_2$, ZnO and $Zn(Ac)_2$ were also checked, but none of them showed superior results to $Zn(ClO_4)_2·6H_2O$ (Table 1, entries 5–9 *versus* entry 4). Results of the screening study of the amount of $Zn(ClO_4)_2·6H_2O$ (Table 1, entries 10–13) indicated that 5 mol% $Zn(ClO_4)_2·6H_2O$ was sufficient for the completion of the reaction within 5 h. Further investigation of the reaction temperature (Table 1, entries 14–18) indicated that 80 °C was optimal for the reaction.

To show the general application of the protocol, and with the optimal conditions in hand (Table 1, entry 17), we then investigated a reactant range of nitriles and halohydrocarbons. As shown in Table 2, the substrate scope of halohydrocarbons under the optimised conditions was firstly examined. Different N-substituted amides were obtained from the reaction of benzonitrile with benzyl, *tert*-butyl and secondary alkyl halohydrocarbons in good to excellent yields. All the reactions proceeded well in 3–5 h with high selectivity without formation of any by-products. We then found that benzyl bromides and chlorides and alkyl bromides and chlorides were all smoothly converted into the corresponding amide products in comparable yields (Table 2, entries 1 and 2, 5 and 7, 12 and 13). Furthermore, benzyl halides bearing either electron-donating or electron-withdrawing substituents (Table 2**,** entries 9–13) afforded the products in similar yields, a result that led us to conclude that the electronic effects do not affect this transformation significantly. Moreover, *tert*-butyl bromide gave *N*-*tert*-butyl amide in 89% (Table 2**,** entry 6). In addition, reactions of *sec*-alkyl halohydrocarbons (cyclopentyl, cyclohexyl and isopropyl halohydrocarbons) were all carried out well and their corresponding products **1c**, **1d** and **1f** were obtained in 85–90% yields.

We next investigated the effect of the structure of the nitrile. As shown in Table 3, it was found that the reactions of a wide variety of benzonitriles, except 2,6-dichlorobenzonitrile (Table 3, entry

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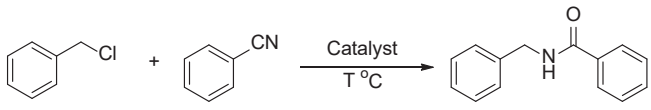
Table 1 Optimisation of reaction conditions[a]

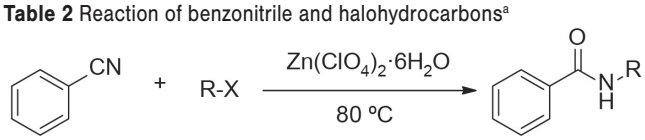


1a

Entry	Catalyst	T (°C)	Amount (%mol)	Time (h)	Yield (%)[b]
1	$Zn(OTf)_2$	100	5	5	53
2	$ZnCl_2$	100	5	5	67
3	$ZnBr_2$	100	5	5	78
4	$Zn(ClO_4)_2{\cdot}6H_2O$	100	5	5	92
5	$Zn(Ac)_2$	100	5	5	18
6	$ZnSO_4$	100	5	5	No reaction
7	ZnI_2	100	5	5	23
8	$Zn(acac)_2$	100	5	5	37
9	ZnO	100	5	5	36
10	$Zn(ClO_4)_2$	100	1	5	68
11	$Zn(ClO_4)_2$	100	2	5	70
12	$Zn(ClO_4)_2$	100	5	5	92
13	$Zn(ClO_4)_2$	100	10	5	92
14	$Zn(ClO_4)_2$	RT	5	5	No reaction
15	$Zn(ClO_4)_2$	40	5	5	No reaction
16	$Zn(ClO_4)_2$	60	5	5	68
17	$Zn(ClO_4)_2$	80	5	5	92
18	$Zn(ClO_4)_2$	100	5	5	92

[a]Benzyl chloride (1.2 mmol), benzonitrile (1 mmol).
[b]Isolated yield.

Table 2 Reaction of benzonitrile and halohydrocarbons[a]



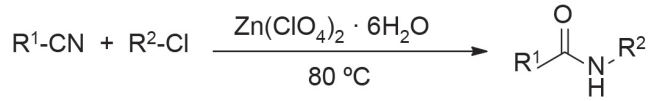
1a–j

Entry	R–X	Time (h)	Yield (%)[b]
1	$PhCH_2Cl$	5	92
2	$PhCH_2Br$	5	92
3	2-$ClPhCH_2Cl$	5	91
4	*cyclo*-C_5H_9Br	4	90
5	IsopropylBr	5	88
6	*tert*-C_4H_9Br	5	89
7	IsopropylCl	5	88
8	*cyclo*-$C_6H_{11}Br$	5	89
9	4-$NO_2C_6H_4CH_2Br$	5	86
10	4-$MeC_6H_4CH_2Br$	3	90
11	2-$MeC_6H_4CH_2Br$	3	94
12	4-$ClC_6H_4CH_2Cl$	3	89
13	4-$ClC_6H_4CH_2Br$	3	89

[a]Halohydrocarbons (1.2 mmol), benzonitrile (1 mmol).
[b]Isolated yield.

11), proceeded well and afforded the corresponding amide products in good to excellent yields. 2,6-Dichlorobenzonitrile was found to be unreactive even with higher catalyst loading (20 mol%), extended reaction time (24 h) and elevated temperature (100–120 °C). This result also appeared in the literature.[15] We also found that electron-donating and electron-withdrawing substituents on the *para*, *ortho* and *meta* sites of the benzonitrile did not hinder the reaction (Table 3**,** entries 2–7). For example, 4-methylbenzonitrile and 4-(trifluoromethyl)benzonitrile gave

Table 3 Reaction of various nitriles and halohydrocarbons[a]



1k–u

Entry	R^1	R^2	Time (h)	Yield (%)[b]
1	3-MeC_6H_4	$PhCH_2$	2	90
2	2-MeC_6H_4	$PhCH_2$	2	89
3	4-MeC_6H_4	$PhCH_2$	2	92
4	4-$F_3CC_6H_4$	$PhCH_2$	2	98
5	3-FC_6H_4	$PhCH_2$	3	92
6	4-$NO_2C_6H_4$	$PhCH_2$	5	86
7	3-BrC_6H_4	$PhCH_2$	2	88
8	4-$MeOC_6H_4$	$PhCH_2$	2	87
9	4-$MeOC_6H_4$	*cyclo*-C_5H_9	3	90
10	4-$F_3CC_6H_4$	*cyclo*-C_6H_{11}	3	94
11	2,6-$Cl_2C_6H_4$	$PhCH_2$	24	No reaction

[a]Nitriles (0.5 mmol) and halohydrocarbons (0.6mmol).
[b]Isolated yields.

the corresponding products **1m** and **1n** in 92 and 98% yields respectively. The results were similar to those from the method reported in the literature,[15] but our method employs shorter reaction times and lower temperatures.

In summary, we have devised an effective and mild protocol for the synthesis of amides from nitriles with halohydrocarbons by employment of $Zn(ClO_4)_2{\cdot}6H_2O$ as an economically effective catalyst under solvent-free conditions. The present work involves several practical advantages like the employment of mild reaction conditions, short reaction times, solvent-free conditions and an easy workup procedure. Especially, we provide another Lewis acid catalyst for the Ritter reaction with halohydrocarbons.

Experimental

All reagents were purchased from commercial sources and used without further purification. Melting points were determined on a RY-1 hot stage microscope and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance DPX-400 MHz instrument in $CDCl_3$. Chemical shifts (δ) are given in part per million (ppm) relative to TMS as an internal standard. All reactions were monitored by thin layer chromatography (TLC) on silica gel GF-254 glass plates (E. Merck) and viewed under UV light at 254 nm.

Reaction of nitriles and halohydrocarbons; typical procedure

CAUTION: $Zn(ClO_4)_2.6H_2O$ can cause skin and serious eye irritation and appropriate care should be taken in its use. In general, perchlorates are explosive compounds and appropriate caution should be taken including the avoidance of evaporation to dryness of perchlorate-containing residues.

A mixture of the nitrile (1 mmol), halohydrocarbon (1.2 mmol) and $Zn(ClO_4)_2{\cdot}6H_2O$ (5 mol%) was placed in a round-bottom flask. Then the reaction mixture was heated at 80 °C for the given time. After completion of the reaction (monitored by TLC), water (5mL) was added to the reaction mixture which was then extracted with ethyl acetate (3 × 5 mL). The organic layers were collected, combined, washed with water (3 × 10 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The pure product was obtained by passing the organic extract through a silica gel (200–300 mesh) column using petroleum ether (60–90 °C)/ethyl acetate (10:1–5:1) as eluent and identified by ^{1}H NMR and ^{13}C NMR. The ^{1}H NMR and

^{13}C NMR spectra of the products are shown in the Electronic Supplementary Information.

N-*Benzylbenzamide* (**1a**): White powder; m.p. 97–98 °C (lit.[15] 96–97 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.80 (m, 2H), 7.78–7.25 (m, 8H), 6.51 (s, 1H), 4.64 (d, *J* = 5.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.4, 138.2, 134.4, 131.6, 128.8, 128.6, 127.9, 127.6, 127.0, 44.1.

N-*(2-Chlorobenzyl)benzamide* (**1b**): White powder; m.p. 113–114 °C (lit.[15] 113–114 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.80–7.24 (m, 9H), 6.68 (s, 1H), 4.74 (d, *J* = 6 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.3, 135.6, 134.3, 133.7, 131.6, 130.4, 129.6, 129.1, 128.6, 127.2, 126.9, 42.1.

N-*Cyclopentylbenzamide* (**1c**): White powder; m.p. 142–143 °C (lit.[15] 143–144 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.76 (m, 2H), 7.50–7.39 (m, 3H), 6.16 (s, 1H), 4.43–4.38 (m, 1H), 2.13–2.05 (m, 2H), 1.75–1.63 (m, 4H), 1.54–1.46 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.2, 134.9, 131.2, 128.5, 126.8, 51.7, 33.2, 23.8.

N-*Isopropylbenzamide* (**1d**): White powder; m.p. 88–89 °C (lit.[15] 89–90 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.77 (m, 2H), 7.49–7.40 (m, 3H), 6.01 (s, 1H), 4.31–4.29 (m, 1H), 1.28–1.26 (m, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 166.7, 134.9, 131.3, 128.5, 126.8, 41.9, 22.9.

N-tert-*Butylbenzamide* (**1e**): White powder; m.p. 118–120 °C (lit.[15] 119–121 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.73 (m, 2H), 7.47–7.39 (m, 3H), 5.98 (s, 1H), 1.48 (s, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 166.9, 135.9, 131.1, 128.5, 126.7, 51.6, 28.9.

N-*Cyclohexylbenzamide* (**1f**): White powder; m.p. 140–141 °C (lit.[15] 139–140 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.77 (m, 2H), 7.51–7.40 (m, 3H), 6.05 (s, 1H), 4.0–3.97 (m, 1H), 2.05–2.02 (m, 2H), 1.74–1.23 (m, 8H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 166.6, 135.1, 131.2, 128.5, 126.8, 48.7, 33.2, 25.6, 24.9.

N-*(4-Nitrobenzyl)benzamide* (**1g**): White powder; m.p. 156–157 °C (lit.[15] 157–158 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.21 (m, 2H), 7.83 (d, *J* = 7.2 Hz, 2H), 7.55–7.45 (m, 5H), 6.70 (s, 1H), 4.77 (d, *J* = 6 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.6, 147.3, 145.9, 133.7, 132.0, 128.8, 128.3, 127.8, 127.0, 43.3.

N-*(4-Methylbenzyl)benzamide* (**1h**): White powder; m.p. 129–131 °C (lit.[15] 129–130 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.79 (m, 2H), 7.49–7.39 (m, 3H), 7.23–7.15 (m, 4H), 6.45 (s, 1H), 4.61 (d, *J* = 5.5 Hz, 2H), 2.45 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.3, 137.4, 135.1, 134.4, 131.5, 129.4, 128.5, 127.9, 126.9, 43.9, 21.1.

N-*(2-Methylbenzyl)benzamide* (**1i**): White powder; m.p. 112–113 °C (lit.[23] 113–114 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.78 (m, 2H), 7.49–7.39 (m, 3H), 7.29–7.19 (m, 4H), 6.33 (s, 1H), 4.63 (d, *J* = 5.2 Hz, 2H), 2.36 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.2, 136.6, 135.7, 134.3, 131.5, 130.6, 128.7, 128.6, 127.9, 126.9, 126.3, 42.3, 19.1.

N-*(4-Chlorobenzyl)benzamide* (**1j**): White powder; m.p. 138–139 °C (lit.[23] 139–140 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.80 (m, 2H), 7.51–7.41 (m, 3H), 7.32–7.28 (m, 4H), 6.60 (s, 1H), 4.61 (d, *J* = 5.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.4, 136.8, 134.1, 133.4, 131.7, 129.2, 128.8, 128.6, 126.9, 43.4.

N-*Benzyl-3-methylbenzamide* (**1k**): White powder; m.p. 98–99 °C (lit.[15] 97–98 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.61–7.55 (m, 2H), 7.34–7.28 (m, 7H), 6.55 (s, 1H), 4.62 (d, *J* = 5.6 Hz, 2H), 2.37 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.6, 138.4, 138.3, 134.3, 132.3, 128.8, 128.4, 127.9, 127.7, 127.6, 123.9, 44.1, 21.3.

N-*Benzyl-2-methylbenzamide* (**1l**): White powder; m.p. 96–98 °C (lit.[15] 98–99 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36–7.15 (m, 9H), 6.16 (s, 1H), 4.61 (d, *J* = 5.8 Hz, 2H), 2.44 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.0, 138.1, 136.2, 133.3, 131.0, 129.9, 128.8, 127.8, 127.6, 126.7, 125.7, 43.9, 19.8.

N-*Benzyl-4-methylbenzamide* (**1m**): White powder; m.p. 133–134 °C (lit.[23] 132–133 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.71 (d, *J* = 8.1 Hz, 2H), 7.36–7.21 (m, 7H), 6.47 (s, 1H), 4.65 (d, *J* = 5.5 Hz, 2H), 2.36 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 167.4, 142.0, 138.3, 131.5, 129.3, 128.7, 127.9, 127.6, 127.0, 44.1, 21.5.

N-*Benzyl-4-(trifluoromethyl)benzamide* (**1n**): White powder; m.p. 168–170 °C (lit.[23] 170–171 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.91 (d, *J* = 8.1 Hz, 2H), 7.71 (d, *J* = 8.1 Hz, 2H), 7.38–7.32 (m, 5H), 6.49 (s, 1H), 4.67 (d, *J* = 5.6 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 166.1, 137.7, 137.6, 133.5 (q, J_{C-F} = 33 Hz), 128.8, 128.0, 127.9, 127.5, 125.6, 125.0 (q, J_{C-F} = 271 Hz), 44.3.

N-*Benzyl-3-fluorobenzamide* (**1o**): White powder; m.p. 91–92 °C (lit.[24] 90.5–91.8 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.54–7.49 (m, 2H), 7.46–7.16 (m, 7H), 6.74 (s, 1H), 4.60 (d, *J* = 5.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 166.3 (q, J_{C-F} = 2 Hz), 163.9 (q, J_{C-F} = 246 Hz), 137.9, 136.6 (q, J_{C-F} = 7 Hz), 130.3 (q, J_{C-F} = 17 Hz), 128.8, 128.4 127.9, 127.7, 122.5 (q, J_{C-F} = 3 Hz), 118.7 (q, J_{C-F} = 21 Hz), 114.6 (q, J_{C-F} = 22 Hz), 44.2.

N-*Benzyl-4-nitrobenzamide* (**1p**): White powder; m.p. 145–146 °C (lit.[15] 144–146 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.28 (d, *J* = 8.8 Hz, 2H), 7.96 (d, *J* = 8.8Hz, 2H), 7.38–7.33 (m, 5H), 6.57 (s, 1H), 4.67 (d, *J* = 5.6 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 165.3, 149.6, 139.9, 137.4, 128.9, 128.2, 128.0, 123.9, 44.5.

N-*Benzyl-3-bromobenzamide* (**1q**): White powder; m.p. 87–88 °C (lit.[23] 87–88 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.92 (s, 1H), 7.70–7.60 (m, 2H), 7.34–7.26 (m, 6H), 6.57 (s, 1H), 4.61 (d, *J* = 5.6 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 166.0, 137.8, 136.3, 134.5, 130.2, 130.1, 128.8, 127.9, 127.7, 125.6, 122.8, 44.3.

N-*Benzyl-4-methoxybenzamide* (**1r**): White powder; m.p. 110–111 °C (lit.[15] 110–112 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.77 (d, *J* = 8.8 Hz, 2H), 7.35–7.28 (m, 5H), 6.91 (d, *J* = 8.8 Hz, 2H), 6.45 (s, 1H), 4.63 (d, *J* = 5.2 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 166.9, 162.2, 138.4, 128.8, 128.7, 127.9, 127.6, 126.6, 113.8, 55.4, 44.1.

N-*Cyclopentyl-4-methoxybenzamide* (**1s**): White powder; m.p. 150–151 °C (lit.[25] 150–152 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.74 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.05 (s, 1H), 4.42–4.37 (m, 1H), 3.85 (s, 3H), 2.11–2.06 (m, 2H), 1.73–1.63 (m, 4H), 1.53–1.45 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 166.7, 162.0, 128.6, 127.2, 113.7, 55.4, 51.6, 33.3, 23.8.

N-*Cyclohexyl-4-(trifluoromethyl)benzamide* (**1t**): White powder; m.p. 168–169 °C (lit.[26] 167–170 °C); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.69 (d, *J* = 8.1 Hz, 2H), 6.08 (d, *J* = 6.2Hz, 1H), 3.99 (m, 1H), 2.06–2.03 (m, 2H), 1.80–1.75 (m, 2H), 1.79–1.70 (m, 1H), 1.45–1.42 (m, 2H), 1.28–1.22 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 165.4, 138.4, 133.5 (q, J_{C-F} = 33 Hz), 127.4, 125.6 (q, J_{C-F} = 4 Hz), 125.1 (q, J_{C-F} = 271 Hz), 49.0, 33.2, 25.5, 24.9.

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Electronic Supplementary Information

The ESI (^{1}H NMR and ^{13}C NMR spectra of the products) is available through

http://ingentaconnect.com/content/stl/jcr/2018/00000042/00000007/art00011

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