A novel method for the conversion of carboxylic acids to *N,N*-dimethylamides using *N,N*-dimethylacetamide as a dimethylamine source

Sanjeev Kumar Aavula*, Anil Chikkulapally, N. Hanumanthappa, Indira Jyothi, C.H. Vinod Kumar, Sulur G. Manjunatha, and Suresh Kumar Sythana

Pharmaceutical Development, AstraZeneca India Pvt. Ltd, off Bellary Road, Hebbal, Bangalore 560 024, India

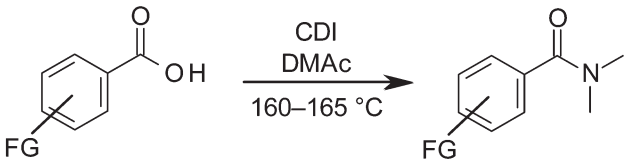
A simple, cost effective and environmentally benign method is reported for the preparation of *N,N*-dimethylamides from carboxylic acids. The versatility of the method is determined by synthesising a large number of *N,N*-dimethylamide derivatives. Carboxylic acids are heated at 160–165 °C in *N,N*-dimethylacetamide solvent in the presence of 1,1′-carbonyldiimidazole to afford the corresponding *N,N*-dimethylamides in good to excellent yields.

Keywords: *N,N*-dimethylacetamide, 1,1′-carbonyldiimidazole, carboxylic acids, amidation, one-pot synthesis

In general, synthetic methods for *N,N*-dimethylamides require the reaction of acid chlorides with *N,N*-dimethylamines in the presence of activating agents. The most typical and industrially used method for the preparation of *N,N*-dimethylbenzamide is the heating of commercially available benzoyl chloride at 150 °C in dimethylformamide (DMF) for a long time.[1] A mixture of DMF and thionyl chloride is known to generate a Vilsmeier complex at 80 °C, and this complex acts as a reagent for chlorination of benzoic acids[2] followed by a *N,N*-dimethylamino group transfer reaction between benzoyl chlorides and DMF at 150 °C. An alternative method for the synthesis of *N,N*-dimethylamides is to use carboxylic acids and dimethyl amine in the presence of coupling reagents.[3] Tetrakis(dimethy lamino)silane in an anhydrous solvent at high temperature has been used for the preparation of *N,N*-dimethylamides from carboxylic acids.[4] A one-pot tandem chlorination and amidation reaction of carboxylic acids in DMF in the presence of thionyl chloride at 150 °C has been reported by Kumagai *et al*.[5] Kaboudin and Haghighat reported a one-pot conversion of carboxylic acids into *N,N*-dimethylamides by using *N,N*-dimethylsulfamoyl imidazole/*N,N*-dimethylsulfamoyl chloride in the presence of a mixture of methanesulfonic acid and phosphorus pentoxide.[6] Recently, Xavier *et al*. Reported solvent-free formation of amide bonds from carboxylic acids and amines using 1,1′-carbonyldiimidazole (CDI).[7]

A few direct methods of *N,N*-dimethylamination of acid chlorides with DMF solvent[1,2] at 150 °C for longer time have been reported in the literature. These processes involve the formation of a Vilsmeier complex (*N,N*-dimethylchloromethyliminium chloride) which is known to be explosive under certain conditions, for example, in contact with metals, and may also undergo decomposition to produce sulfur, dimethylcarbamoylchloride (a potent animal carcinogen) and liberation of toxic gases like sulfur dioxide (from thionyl chloride) and carbon monoxide (from the DMF solvent).

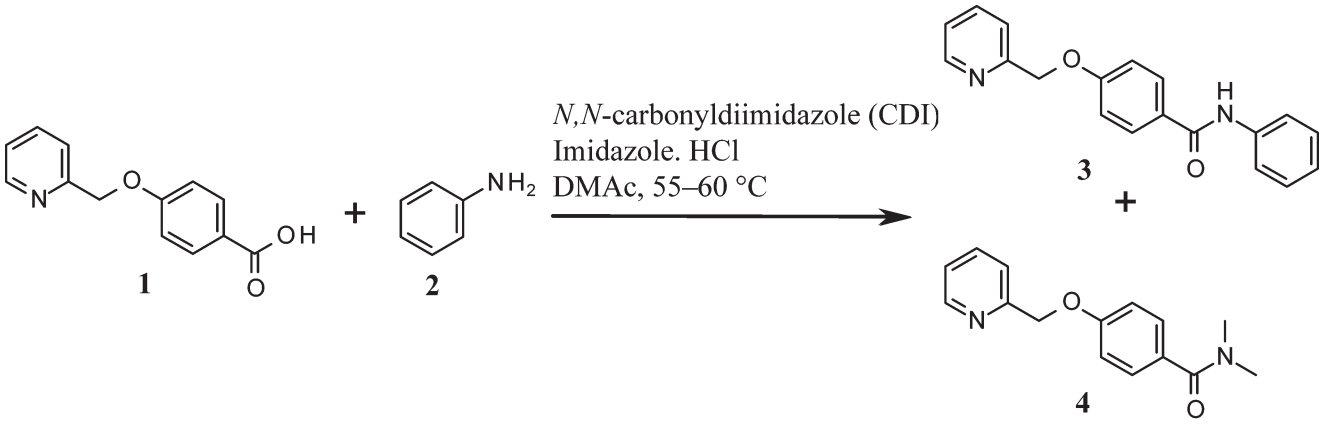
We report here a safe, efficient and novel method for the *N,N*-dimethylamination of carboxylic acids (Scheme 1). Carboxylic acids were heated at 160–165 °C in *N,N*-dimethylacetamide solvent in the presence of CDI to afford the corresponding *N,N*-dimethylamides in good to excellent yields in short times. To the best of our knowledge, there is no report in the literature that DMAc solvent in combination with CDI can be used as a source of *N,N*-dimethylamine. The advantages of this method are that the by-products are carbon dioxide, *N*-acetyl imidazole and imidazole which are not especially hazardous; in addition to this the imidazole and *N*-acetyl imidazole by-products can be washed out by simple water washes.



Scheme 1 Synthesis of *N,N*-dimethylamides where FG=CN, $N(CH_3)_2$, F, Br, Me, $CH=CH_2$, $COCH_3$, COOH, OMe.

Results and discussion

We wished to synthesise benzamide **3**, which we planned to do by coupling of acid **1** and aniline **2** with CDI mediated amide coupling in dimethylacetamide (DMAc) solvent at 55–60 °C for 120 mins (Scheme 2), but were surprised to find **4** (15–20% area by HPLC) along with product **3**. This finding encouraged us to study the scope and limitations of DMAc as a potential



Scheme 2 CDI-mediated amidation in DMAc solvent.

* Correspondent. E-mail: Sanjeev.aavula@astrazeneca.com

N,N-dimethyl amine precursor for amidation reaction in more detail.

First, we attempted to synthesise *N,N*-dimethylbenzamide **7** from benzoic acid **5** in DMAc solvent without an external amine source to assess whether one pot *N,N*-dimethylamination is accessible in the presence of the CDI reagent. Thus, benzoic acid (1.0 g, 0.0081 mol) and CDI (2.0 g, 0.012 mol) were heated to 160–165 °C in dry *N,N*-dimethylacetamide (10 mL) solvent. The benzamide **7** could be isolated in 82% yield after a reaction time of 1 h (Scheme 3).

To investigate the mechanism of the reaction a series of experiments were screened with isolated imidazolide intermediate **6** (*N*-benzoylimidazole), and the results are presented in Table 1. The amidation reaction proceeded smoothly in the presence of CDI/imidazole (entries 1–3). The reaction is much faster with CDI reagent as compared to imidazole. Attempts to conduct the amidation reaction only with DMAc solvent in the absence of both CDI and imidazole (entry 4), did not produce amidation product **7** and the starting material was recovered almost quantitatively by column chromatography.

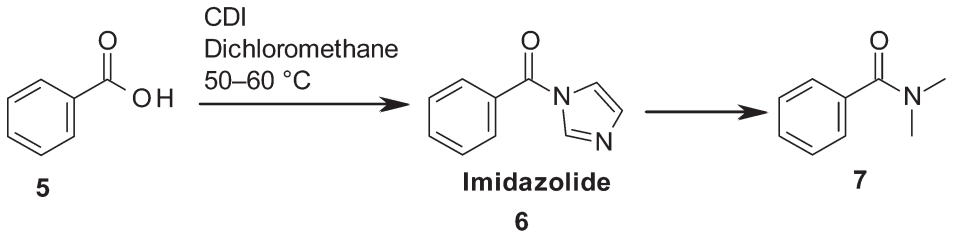
A reaction sample was withdrawn for gas chromatography mass spectrometric analysis (GCMS) after 1 h. Two peaks corresponding to mass 110 and 149 were observed, which corresponded to the structures of *N,N*-Dimethylbenzamide (**7**) and *N*-acetyl imidazole respectively (Figs 1, 2 and 3).

Table 1 Amidation of imidazolide **6** under different reaction conditions

Entry	Substrate	DMAc /mL	CDI/mol equiv.	Imidazole /mol equiv.	Time /h	Purity by HPLC **7**/%
1	**6**	10	1.0	–	2.0	85
2	**6**	10	0.25	–	4.0	84
3	6	10	–	1.0	24.0	66
4	**6**	10	–	–	6.0	0

Reaction conditions: imidazolide **6** (0.05 moles), CDI (0.05/0.0015 moles), imidazole (0.05 moles), and heating (160–165 °C, 2–24 h).

Based on the above optimisation work (Table 1) and GC-MS analysis, the reaction mechanism (Scheme 4) for the formation of *N,N*-dimethylamides may be suggested; nucleophilic attack of imidazole on DMAc results in the formation of tetrahedral intermediate **A**. Attack on the tetrahedral intermediate by imidazole leads to the generation of *N*-acetyl imidazole and *N,N*-dimethylamine. So it is then possible that the liberated



Scheme 3 *N,N*-Dimethylamination of benzoic acid.

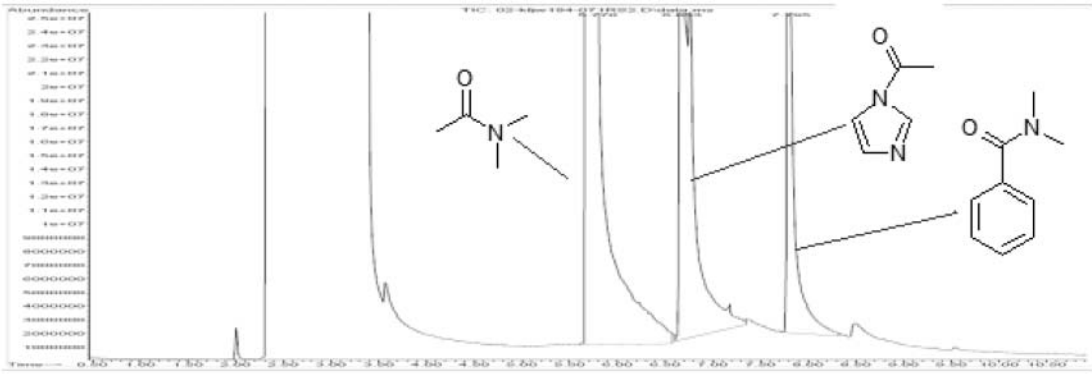


Fig. 1 GC trace of *N,N*-dimethylamination of benzoic acid.

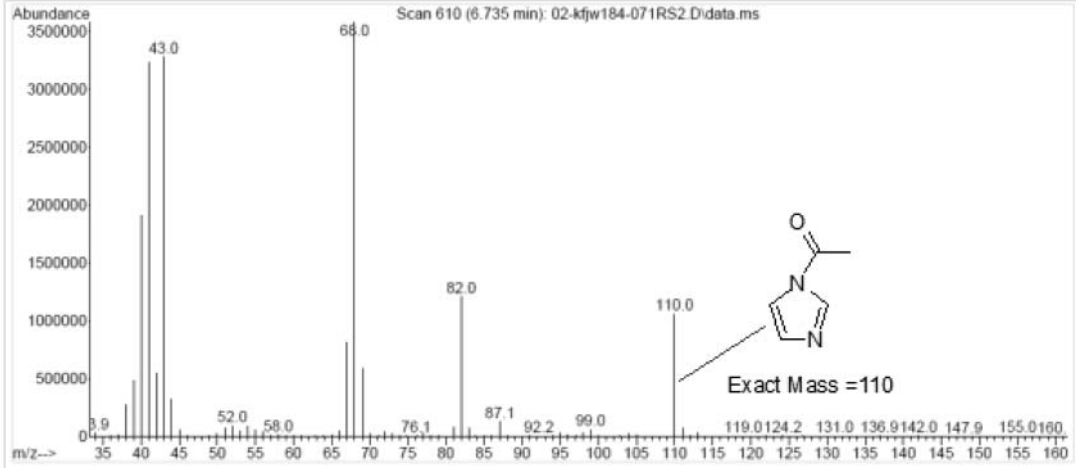


Fig. 2 GC-MS of *N*-acetyl imidazole.

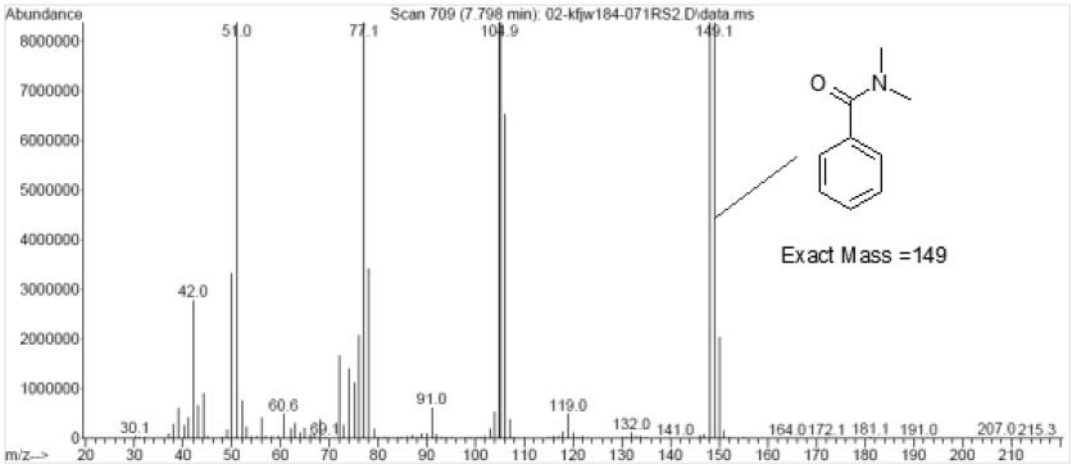


Fig. 3 GC-MS of *N,N*-dimethylbenzamide (**7**).

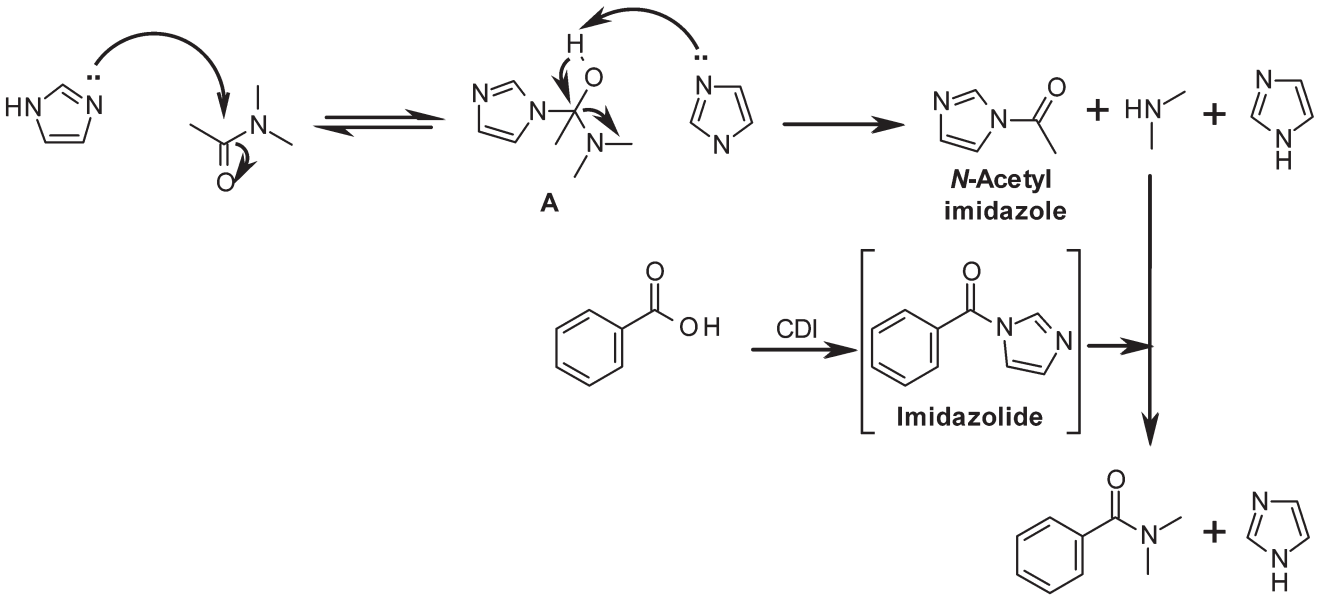
dimethylamine reacts with the reactive species imidazolide to yield the amide.

The dimethylamination of benzoic acid (Table 2, entry 12) using DMAc and CDI was the first *N,N*-dimethylamination reaction to be investigated. Benzoic acid was added to 2.0 equiv. of CDI in DMAc solvent and heated at 160–165 °C for 1.5 h. HPLC analysis of the crude reaction product showed that the reaction had proceeded to 100% conversion. The reaction mixture could be readily purified by extracting the product in isopropyl acetate solvent followed by water washes to remove the by-products. The solvent was then concentrated under reduced pressure to give an analytically pure sample of *N,N*-dimethylbenzamide (Table 2, entry 12) in 82% yield without the need for column chromatography. This successful dimethylamination protocol was then applied to a range of carboxylic acids, with the results summarised in Table 2.

The dimethylamination reactions of benzoic acid derivatives (Table 2, entries 1–7, 9, 10, 12, 15 and 16) proceeded well, enabling the *N,N*-dimethylamides products to be isolated above 80% yields, except for 4-formylbenzoic acid where product formation was observed. Here decomposition had occurred under the conditions for proposed 4-formyl-*N,N*-dimethylbenzamide preparation (Table 2, entry 17) leading to the multiple spots in TLC. The amination reaction of a disubstituted acid substrate (Table 2, entry 14) was examined under the conditions. The yield of phthalamide was relatively low (< 10%) probably due to poor solubility of the acids in DMAc. When the reaction times were prolonged, the yields went up to 80%. Due to poor solubility of the product in isopropyl acetate solvent, this was replaced by the highly polar solvent n-butanol for extraction of the product. The reaction even worked well for five membered heterocycles. For example, 2-furonoic acid gave *N,N*-dimethylfuran-2-carboxamide (Table 2, entry 8) in 64% yield. Amination of 1-naphthalenecarboxylic acid (Table 2, entry 10) successfully produced *N,N*-dimethyl-1-naphthamide in good yield. The reaction equally worked for the aliphatic acids (Table 2, entries 13, 18 and 19) that were examined under the conditions. Amination proceeded smoothly to afford corresponding *N,N*-dimethylamide products in 70–76% yields.

Conclusions

In conclusion, DMAc in combination with CDI provides a simple, mild, good yield and relatively clean reaction for the synthesis of *N,N*-dimethylamides. This method is an attractive



Scheme 4 A proposed mechanism of *N,N*-dimethylamination of carboxylic acid.

Table 2 Conversion of carboxylic acids to *N,N*-dimethylamides using DMAc and CDI at 160–165 °C

Entry	R	Product	Time/h	Yield/%[a]
1	*p*-NCC_6H_4	**9a**	1	85
2	*p*-$(CH_3)_2NC_6H_4$	**9b**	1	80
3	*o*-FC_6H_4	**9c**	1	82
4	3,5-$(CH_3)_2C_6H_3$	**9d**	1	80
5	*p*-ClC_6H_4	**9e**	1	90
6	*p*-CH_2=CHC_6H_4	**9f**	1.5	70
7	*p*-$CH_3COC_6H_4$	**9g**	1	87
8	2-Furyl	**9h**	2	64
9	*o*-$CH_3OC_6H_4$	**9i**	1	85
10	1-Naphthy	**9j**	1	90
11	*m*-BrC_6H_4	**9k**	1	89
12	C_6H_5	**9l**	1.5	82
13	*p*-ClC_6H_4-CH_2	**9m**	0.5	70
14	1,4-$(COOH)_2C_6H_4$	**9n**	2	80
15	*p*-$CH_3C_6H_4$	**9o**	1	85
16	*p*-(2-C_5H_4N-CH_2)-O-C_6H_4	**9p**	1	90
17	*o*-$OHCC_6H_4$	**9q**	0.5	–
18	C_6H_5-CH_2CH_2	**9r**	0.5	76
19	*p*-$CH_3OC_6H_4$-CH_2	**9s**	0.5	72

[a] Yields refer to the isolated pure product.

and useful contribution to present methodologies for the conversion of carboxylic acids into corresponding amides. This protocol is green as it has several advantages over the previous methods reported which include (i) shorter reaction times and easy workup, (ii) *in-situ* generation of dimethylamine, thereby simplifying the operation, (iii) highly water soluble by-products, and (iv) excellent chemical yields for wide variety of substrates.

Experimental

All reagents were purchased from commercial sources and used without any additional purification. All experiments were carried out under a nitrogen atmosphere to maintain anhydrous conditions. ^{1}H NMR and ^{13}C NMR spectra were recorded on a 400-MHz Bruker instrument with the chemical shifts reported as δ ppm and couplings expressed in Hertz. The chemical shifts, δ, were recorded relative to tetramethylsilane as an internal standard; all coupling constants, *J*, are reported in Hz.

*Synthesis of N-benzoylimidazole (**6**); typical procedure*

1,1′-Carbonyldiimidazole (2.0 g, 0.012 mol) was added to *a* solution of benzoic acid (**5**) (1.0 g, 0.0081 mol) in dichloromethane (6 mL). The solution was stirred for 1.5 h. Dichloromethane (20 mL) was added to the reaction mixture followed by water (40 mL). The dichloromethane layer was separated and dried over anhydrous Na_2SO_4 and filtered. Distillation of the dichloromethane extract gave 1.3 g (93%) of *N*-benzoylimidazole (**6**) as a colourless pasty liquid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (s, 1H) 7.76–7.69 (m, 1H), 7.56–7.53 (m, 2H), 7.46–7.42 (m, 1H) 7.33–7.29 (m, 2H), 6.91 (s, 1H).

N,N-dimethylamination of acids; typical procedure

A solution of benzoic acid (Table 2, entry 12) (1.0 g, 0.0081 mol) and 1,1′-carbonyldiimidazole (2.0 g, 0.012 mol) in dry *N,N*-dimethylacetamide (10 mL) solvent was heated to 160–165 °C and stirred for 1 h. HPLC analysis of the crude product showed that the reaction had proceeded to 100% conversion. The resultant solution was cooled to 20–25 °C and quenched with water (30 mL). The reaction mixture was extracted with isopropyl acetate (2 × 15 mL). The combined organic layer was washed with 5% aqueous NaOH solution (2 × 15 mL), dried over anhydrous Na_2SO_4 and filtered. The filtrate was evaporated under reduced pressure to give 0.98 g (82%) of *N,N*-dimethylbenzamide (Table 2, entry 12).

All experiments were run on a 1.0 g scale using dry *N,N*-dimethylacetamide under nitrogen atmosphere with 1,1′-carbonyldiimidazole. The yields refer to isolated yields as such and/ or recrystallisation. All products gave satisfactory analytical data. Identification of the products was carried out by ^{1}H NMR, ^{13}C NMR and mass spectroscopic analytical techniques.

N,N-Dimethyl-4-cyanobenzamide (**9a**):[5] Yield (85%); colourless solid: m.p. 98–100 °C (lit.[5] 99–100 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76–7.67 (d, *J* = 8.5 Hz, 2H), 7.57–7.49 (d, *J* = 8.5 Hz, 2H), 3.13 (s, 3H), 2.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.3, 140.6, 132.2, 127.5, 118.1, 113.0, 39.2, 35.1.

N,N-Dimethyl-4-dimethylaminobenzamide (**9b**):[5] Yield (80%); brown solid; m.p. 88–90 °C (lit.[5] 89–90 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41–7.34 (m, 2H), 6.70–6.63 (m, 2H), 3.07 (s, 6H), 2.99 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172, 151.2, 129.1, 122.9, 110.9, 40.1, 38.5.

N,N-Dimethyl-2-fluorobenzamide (**9c**): Yield (82%); colourless waxy solid; ^{1}H NMR (400 MHz, CDCl3): δ 7.45–7.33 (m, 2H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.13–7.09 (m, 1H), 3.13 (s, 3H), 2.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7, 158.1 (d, C, *J* = 246 Hz), 131.2, 128.9, 124.6, 115.8, 115.5, 38.9, 34.9; HRMS (ESI): *m/z* [M+H$^+$] Calcd for $C_9H_{11}FNO$: 168.0825; found: 168.0836.

N,N-Dimethyl-3,5-dimethylbenzamide (**9d**): Yield (80%); colourless waxy solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.06–6.97 (m, 3H), 3.17–3.03 (s, 3H), 2.96 (s, 3H), 2.32 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.0, 137.9, 136.3, 130.9, 124.5, 39.5, 35.2, 21.2. HRMS (ESI): *m/z* [M+H$^+$] Calcd for $C_{11}H_{16}NO$: 178.1231; found: 178.1242.

N,N-Dimethyl-4-chlorobenzamide (**9e**):[6] Yield (90%); colourless solid: m.p. 56–58 °C (lit.[6] 56–57 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44–7.32 (m, 4H), 3.10 (br, s, 3H), 2.96 (br, s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.1, 135.2, 134.4, 128.33, 128.28, 39.2, 35.1.

N,N-Dimethyl-4-ethenylbenzamide (**9f**):[9,11] Yield (70%); colourless solid: m.p. 53–54 °C (lit.[11] 55–56 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50–7.34 (m, 4H), 6.72 (dd, *J* = 17.6, 10.5 Hz, 1H), 5.79 (d, *J* = 17.6 Hz, 1H), 5.31 (d, *J* = 10.5 Hz, 1H), 3.10 (br, s, 3H), 2.98 (br, s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.6, 139.0, 136.4, 135.8, 127.7, 126.3, 115.3, 39.8, 35.6.

N,N-Dimethyl-4-acetylbenzamide (**9g**):[8] Yield (87%); brownish viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04–7.96 (d, *J* = 8.5 Hz, 2H), 7.55–7.49 (d, *J* = 8.5 Hz, 2H), 3.13 (s, 3H), 2.96 (s, 3H), 2.63 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.4, 170.5, 140.7, 137.6, 128.4, 127.2, 39.3, 35.6, 26.7.

N,N-Dimethylfuran-2-carboxamide (**9h**):[10] Yield (64%); colourless viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57–7.45 (m, 1H), 6.99 (d, *J* = 4.0 Hz, 1H), 6.48 (dd, *J* = 4.0 Hz, 1H), 3.28 (br, s, 3H), 3.10 (br, s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.3, 148.0, 143.7, 116.0, 111.1, 38.9, 36.2.

N,N-Dimethyl-2-methoxybenzamide (**9i**):[10] Yield (85%); colourless viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39–7.30 (m, 1H), 7.23 (dd, 8.0 Hz, 1H), 6.98 (t, *J* = 8.0 Hz, 1H), 6.91 (d, *J* = 8.0 Hz, 1H), 3.83 (s, 3H), 3.12 (s, 3H), 2.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.4, 155.2, 130.2, 127.8, 126.2, 120.8, 110.9, 55.5, 38.1, 34.6.

N,N-Dimethyl-1-naphthamide (**9j**):[10] Yield (90%); colourless viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92–7.82 (m, 3H), 7.82–7.45 (m, 4H), 3.25 (s, 3H), 2.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8, 134.6, 133.3, 129.3, 128.9, 128.3, 126.9, 126.2, 125.1, 124.7, 123.8, 38.8, 34.8.

N,N-Dimethyl-3-bromobenzamide (**9k**): Yield (89.0%); colourless viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61–7.49 (m, 2H), 7.38–7.31 (m, 1H), 7.31–7.24 (m, 1H), 3.10 (s, 3H), 2.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 169.7, 138.2, 132.4, 129.9, 125.5, 122.3, 39.4, 35.2. HRMS (ESI): *m/z* [M+H$^+$] Calcd for $C_9H_{11}N^{79}BrO$: 228.0018; found: 228.0037.

N,N-Dimethylbenzamide (**9l**):[5] Yield (82%); colourless solid: m.p. 39–40 °C (lit.[5] 38–39 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46–7.33 (m, 5H), 3.11 (br, s, 3H), 3.04–2.88 (s, 3H); ^{13}C (100 MHz, $CDCl_3$): 171.6, 136.3, 129.5, 128.3, 127.0, 39.5, 35.3.

N,N-Dimethyl-4-chlorophenylacetamide (**9m**): Yield (70%); waxy solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (dd, *J* = 2.0, 7.0 Hz, 1H), 7.32–7.26 (m, 1H), 7.26–7.16 (m, 2H), 3.81 (s, 2H), 3.04 (s, 3H), 3.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 170.1, 133.9, 133.5, 130.7, 129.3, 128.2, 126.9, 38.1, 37.5, 35.6; HRMS (ESI): *m/z* [M+H$^+$] Calcd for $C_{10}H_{13}{}^{35}ClNO$: 198.06816; found: 198.0693.

N,N,N′,N′-Tetramethylterephthalamide (**9n**): Yield (80%); colourless solid: m.p. 145–146 °C (lit.[5] 145–146 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (s, 4H), 3.12 (br s, 6H), 2.97 (br s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8, 137.5, 126.8, 39.5, 35.3.

N,N-Dimethyl-4-methylbenzamide(**9o**):[10] Yield (85%); colourless viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35–7.27 (m, 2H), 7.23–7.15 (m, 2H), 3.09 (br, s, 3H), 2.98 (br, s, 3H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.8, 139.6, 133.3, 128.9, 127.1, 39.3, 35.1, 21.3.

N,N-Dimethyl-4-(pyridine-2-yl-methoxy)benzamide (**9p**): Yield (90%); waxy brownish solid; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.59 (d, *J* = 4.0 Hz, 1H), 7.98–7.74 (m, 1H), 7.53 (d, *J* = 8.0, 1H), 7.43–7.27 (m, 3H) 7.06 (d, *J* = 8.0 Hz, 2H), 5.22 (s, 2H), 2.94 (s, br, 6H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 169.8, 158.8, 156.3, 149.1, 136.9, 128.9, 128.8, 123.0, 121.7, 114.2, 70.3, 40.1, 38.6. HRMS (ESI): *m/z* [M+H$^+$] Calcd for $C_{15}H_{17}N_2O_2$: 257.1285; found: 257.1293.

N,N-Dimethyl-3-phenylpropanamide (**9r**):[12] Yield (76%); brownish viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (m, 2H), 7.24–7.16 (m, 3H), 2.97 (t, *J* = 8.2 Hz, 2H), 2.95 (s, 3H), 2.93 (s, 3H), 2.62 (t, *J* = 8.2 Hz, 2H); ESIMS: *m/z* Calcd [M+]: 177.1; found: 178.1 [M+1].

N,N-Dimethyl-2-(4-methoxyphenyl)acetamide (**9s**):[13] Yield (72%); brownish oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 8.5 Hz, 2H), 3.76 (s, 3H), 3.65 (s, 2H), 2.99 (s, 3H), 2.95 (s, 3H); ESIMS: *m/z* Calcd [M+]: 193.1; found: 194.1 [M+1].

We thank Sudhir Nambiar, R. Sridharan, Bill Moss and Ramachandra Puranik for useful discussions and for help with the manuscript preparation.

Received 20 October 2012; accepted 9 January 2013
Paper 1201581 doi: 10.3184/174751913X13602686612047
Published online: 12 March 2013

References

1 G.M. Coppinger, *J. Am. Chem. Soc.*, 1954, **76**, 1372.
2 H.H. Bosshard, R. Mory, M. Schmidt and H. Zollinger, *Helv. Chim. Acta*, 1959, **42**, 1653.
3 J.I. Le and H.A. Park, *Bull. Kor. Chem. Soc*., 2001, **22**, 421.
4 R.A. Guobin, *Chin. J. Org. Chem.*, 1996, **16**, 68.
5 T. Kumagai, T. Anki, T. Ebi, A. Konishi, K. Matsumoto, H. Kurata, T. Kubo, K. Katsumoto, C. Kitamura and T. Kawase, *Tetrahedron*, 2010, **66**, 8968.
6 B. Kaboudin and H. Haghighat, *J. Iran. Chem. Soc*. 2012 (in press).
7 M.T. Xavier, B. Julien, R. Thomas, S. Jordi, M. Jean and L. Frederic, *Chem. Commun.*, 2012, **48**, 11781.
8 D.N. Sawant, Y.S. Wagh, K.D. Bhatte and B.M. Bhanage, *J. Org. Chem*., 2011, **76**, 5489.
9 S.E. Denmark and C.R. Butler, *J. Am. Chem. Soc.*, 2008, **130**, 3690.
10 Z. Liu, J. Zhang, S. Chen, E. Shi, Y. Xu and X. Wan, *Angew. Chem.*, *Int. Ed.*, 2012, **51**, 3232.
11 T. Ishizone, S. Wakabayashi, A. Hirao, and S. Nakahama, *Macromol.*, 1991, **24**, 5015.
12 Y. Sunada, H. Kawakami, T. Imaoka, Y. Motoyama and H. Nagashima, *Angew. Chem.*, *Int. Ed.*, 2009, **48**, 9511.
13 K.H. Shaughnessy, B.C. Hamann and J.F. Hartwig, *J. Org. Chem*., 1998, **63**, 6546.