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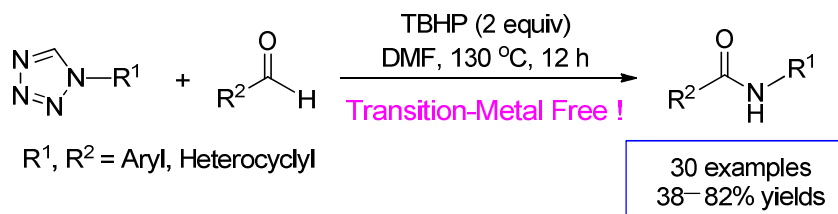
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Synthesis of Amides through an Oxidative Amidation of Tetrazoles with Aldehydes under Transition-Metal-Free Conditions

Juan Du, Kai Luo, and Xiuli Zhang*

Abstract: An inexpensive and efficient synthesis of amides was developed via directly oxidative amidation of tetrazoles with aldehydes under transition-metal-free conditions.



Department of Chemistry, Anhui Agricultural University, Hefei, Anhui 230036, P R China; E-mail: zhxiuli@163.com

Tel.: +86-551-6578-6791

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ARTICLE

Synthesis of Amides through an Oxidative Amidation of Tetrazoles with Aldehydes under Transition-Metal-Free Conditions†

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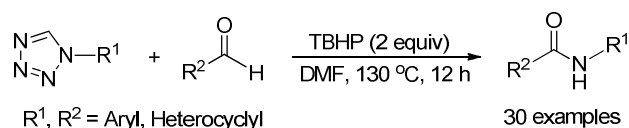
DOI: 10.1039/b000000x

Abstract: A simple, inexpensive and efficient one-pot synthesis of amides was achieved in good yields via the directly oxidative amidation of tetrazoles with aldehydes under transition-metal-free conditions.

Amides have attracted considerable attention not only because of their remarkable biological and pharmacological activity, but also because of they are useful functional groups for the preparation of various organic compounds.¹⁻² To our knowledge, more than 50% of known drugs contain an amide group.³ Amide formation reaction is one of the key cornerstone reactions in organic chemistry.⁴ Typically, amide bond is synthesized by acylation of amines with carboxylic acid derivatives (acid chloride, anhydride, active esters, etc.).⁵ It is estimated that amide formation accounts for 16% of all reactions are used in the synthesis of current pharmaceuticals.³ However, these strategies have several innate drawbacks, such as using highly hazardous reagents, poor atom-efficiency. To circumvent these problems, alternative methods for the synthesis of amide were developed, such as Staudinger reaction,⁶ Schmidt reaction,⁷ Beckmann rearrangement,⁸ aminocarbonylation of haloarenes,⁹ iodonium-promoted α -halo nitroalkane amine coupling,¹⁰ direct amide synthesis from alcohols with amines or nitroarenes,¹¹ hydroamination of alkynes,¹² amidation of thioacids with azides,¹³ and transamidation of primary amides.¹⁴ Unfortunately, most of the methods outlined above have not been applied in industry due to drawbacks such as the use of expensive transition metal catalysis,¹⁵ limited substrate scope, harsh reaction conditions, and growing focus on green chemistry,¹⁶ etc. Hence, the development of efficient and practical amide formation reactions remains a great challenge. In the past decades, great efforts have been made to develop environmentally and friendly methods to amides synthesis.¹⁷ Among the emerging amide formation methods, transition-metal-free oxidative amidation is an attractive method with potential industrial applications.

Apart from the wide applications of 1-aryltetrazoles in rocket propellants and explosives, they are also used for the

organic transformation via their C–H bond functionalizations.¹⁸ To the best of our knowledge, there are no examples using *tert*-butyl hydroperoxide system for the direct oxidative amidation with aldehydes under transition-metal-free conditions. It can overcome the drawbacks of the expensive, poisonous, and air-sensitive properties of metals or organometallics. Herein, the reaction of tetrazoles with aldehydes for direct synthesis of amides in the presence of *tert*-butyl hydroperoxide (TBHP) will be described, which generated the desired products in good yields (Scheme 1).

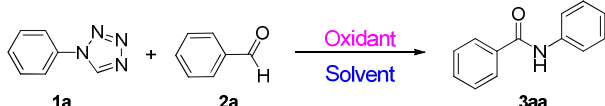


Scheme 1 The amidation of tetrazoles with aldehydes

In our initial attempt, we chose the reaction of 1-phenyltetrazole with benzaldehyde as the model substrates. The results on the reaction conditions screening were shown in Table 1. It could be found that *tert*-butyl hydroperoxide (TBHP, 70% aqueous solution, 2.0 equiv) was favored as the best oxidant for the model reaction in DMF (*N,N*-dimethylformamide), providing desired product **3aa** in 82% yield (Table 1, entry 1). Other oxidants, such as CHP (cumene hydroperoxide), DTBP (di-*tert*-butyl peroxide), H₂O₂ and PhI(OAc)₂ were less effective and gave **3aa** in 48–62% yields (Table 1, entries 2–5). However, it was found that K₂S₂O₈, (NH₄)₂S₂O₈, and I₂ obviously shut down the transformation completely (Table 1, entries 6–8). The solvent is also plays an important role in the reaction. Among the tested solvents, DMF (*N,N*-dimethylformamide) was the best one in the model reaction (Table 1, entry 1). On performing the model reaction in DCE (1,2-dichloroethane) and THF (tetrahydrofuran) afforded **3aa** in 40% and 32% yield, respectively (Table 1, entries 9 and 10). However, the reaction did not work in DMSO (dimethyl sulfoxide), NMP (*N*-methylpyrrolidone), CH₃NO₂, toluene, CH₃CN, EtOH, CH₂Cl₂, acetone, EtOAc, DMAC (dimethylacetamide), 1,4-dioxane, or H₂O (Table 1, entries 11–22).

Department of Chemistry, Anhui Agricultural University, Hefei, Anhui 230036, P R China; E-mail: zhixuli@163.com
Tel.: +86-551-6578-6791

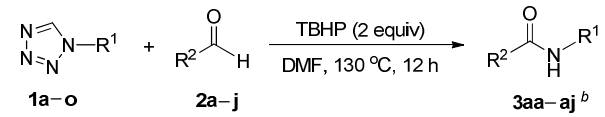
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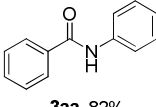
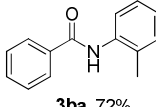
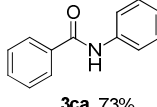
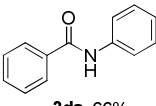
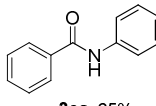
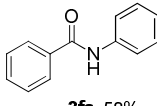
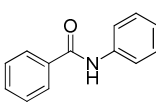
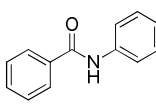
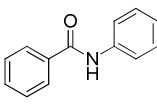
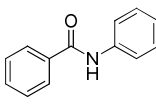
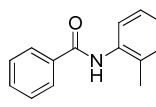
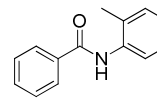
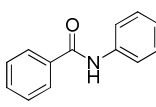
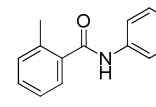
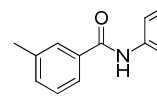
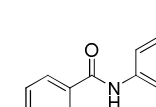
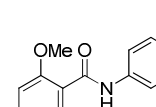
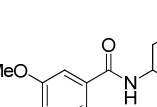
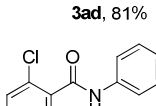
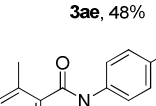
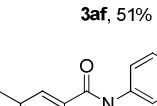
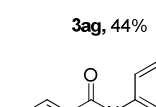
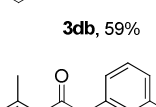
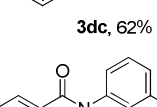
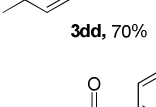
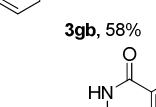
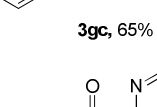
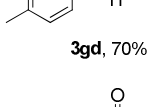
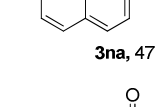
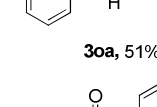
Table 1. Optimization of the reaction conditions for the oxidative amidation of **1a** with **2a**.^a


Entry	Oxidant	Solvent	T/°C	Yield ^b (%)
1	TBHP	DMF	130	82
2	CHP	DMF	130	62
3	DTBP	DMF	130	59
4	H ₂ O ₂	DMF	130	55 ^c
5	PhI(OAc) ₂	DMF	130	48
6	K ₂ S ₂ O ₈	DMF	130	N. R.
7	(NH ₄) ₂ S ₂ O ₈	DMF	130	N. R.
8	I ₂	DMF	130	N. R.
9	TBHP	DCE	80	40
10	TBHP	THF	70	32
11	TBHP	DMSO	130	N. R.
12	TBHP	NMP	130	N. R.
13	TBHP	CH ₃ NO ₂	100	N. R.
14	TBHP	Toluene	110	N. R.
15	TBHP	CH ₃ CN	80	N. R.
16	TBHP	EtOH	80	N. R.
17	TBHP	CH ₂ Cl ₂	50	N. R.
18	TBHP	Acetone	60	N. R.
19	TBHP	EtOAc	80	N. R.
20	TBHP	DMAC	130	N. R.
21	TBHP	1,4-Dioxane	100	N. R.
22	TBHP	H ₂ O	100	N. R.
23	TBHP	DMF	130	61 ^d
24	TBHP	DMF	130	45 ^e
25	TBHP	DMF	130	27 ^f

^a Reaction conditions: **1a** (0.50 mmol), **2a** (1.0 mmol), oxidant (2.0 equiv), sealed tube, solvent (2.0 mL) at the temperature indicated in Table 1 for 12 h, unless otherwise noted, TBHP (70% aqueous) was used. N. R. = No reaction.
^b Isolated yield.
^c 35% wt in H₂O.
^d **2a** (0.75 mmol, 1.5 equiv) was used.
^e **2a** (0.50 mmol, 1.0 equiv) was used.
^f **2a** (0.25 mmol, 0.50 equiv) was used.

For the investigation of the molar ratio between **1a** and **2a**, we found that the molar ratio of **2a/1a** more than 2.0 gave the best yield of **3aa**. When the molar ratio of **2a/1a** is less than 2.0, the reaction generated the poor yields of product **3aa** (Table 1, entries 23–25). During the course of further optimization of the reaction conditions, the reaction was generally completed within 12 h when it was performed in DMF at 130 °C by using TBHP (70% aqueous solution, 2.0 equiv) as sole oxidant.

Table 2. Oxidative amidation of tetrazoles with aldehydes.^a


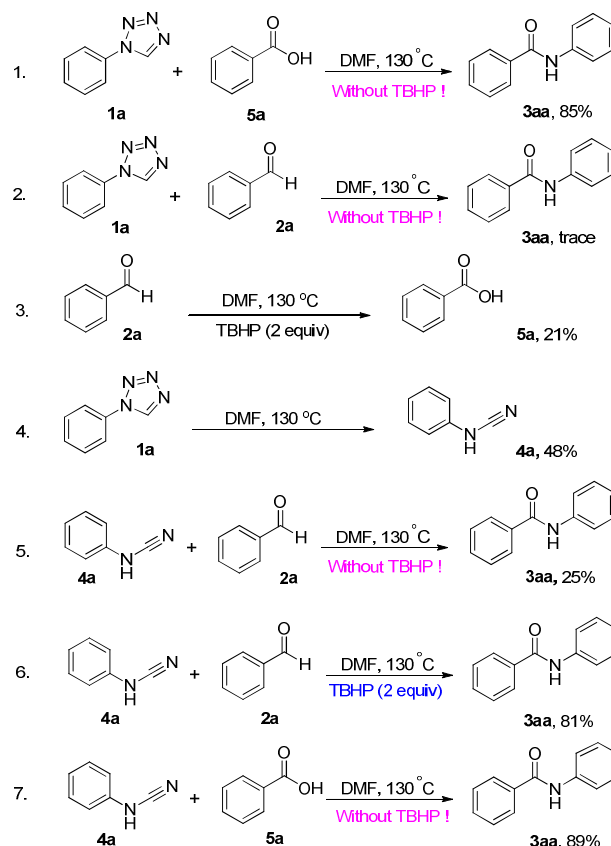
		
		
		
		
		
		
		
		
		
		

^a Reaction conditions: **1** (0.50 mmol), **2** (1.0 mmol), TBHP (70% aqueous solution, 2.0 equiv), DMF (2.0 mL), 130 °C, 12 h.
^b Isolated yields.

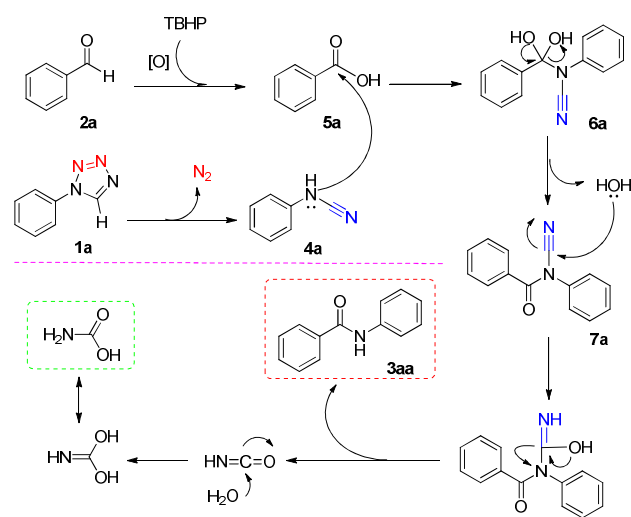
After the optimized reaction conditions had been established, the methodology on the reaction of an array of substituted tetrazoles and commercially available aldehydes was examined (Table 2). As shown in Table 2, we found that a wide range of substituted tetrazoles were suitable for the reaction. 1-Aryltetrazoles with both electron-donating groups (Me, Et, *i*-Pr, MeO, EtO) and an electron-withdrawing group (F) on the benzene rings could react with benzaldehyde smoothly to generate the desired products **3aa–3ma** in 53–82% yields (Table 2). Furthermore, substituents at different positions of the benzene rings in 1-aryltetrazoles (*para*-, *meta*-, or *ortho*-position) did not affect the efficiency of the reaction obviously (Table 2, **3ba–3da**, **3ka–3ma**). The scope of this metal-free amidation was further expanded to a variety of aldehydes. Arylaldehydes attached with electron-donating groups (Me, MeO) or an electron-withdrawing group (Cl) on the benzene rings reacted with 1-aryltetrazoles to afford the corresponding products **3ab–3gd** in 44–81% yields (Table 2). It should be noted that an obvious *ortho*-position effect was observed in the reaction of arylaldehydes with tetrazoles (Table 2, **3ab**, **3ae**, **3ag**, **3db**, **3gb**). For example, *ortho*-methylbenzaldehyde reacted with 1-phenyltetrazole to give the anticipated product **3ab** in 58% yield; but *para*-methylbenzaldehyde reacted with 1-phenyltetrazole to afford the desired product **3ad** in 81% yield. It is important to note that the reactions of 1-(naphthalen-1-yl)-1*H*-tetrazole with benzaldehyde, and 1-phenyltetrazole with 2-naphthaldehyde underwent to generate the corresponding products **3na** and **3ah** in 47% and 55% yield, respectively. Meanwhile, 2-(1*H*-tetrazol-1-yl)pyridine reacted with benzaldehyde, providing the product **3oa** in 51% yield. Heterocyclic aldehydes, such as quinoline-2-carbaldehyde and thiophene-2-carbaldehyde also reacted with 1-phenyltetrazole to form the desired products **3ai** and **3aj** in 49%, 38% yield, respectively (Table 2). However, when aliphatic aldehyde, such as butyraldehyde or heptanal reacted with 1-phenyl-1*H*-tetrazole under the standard reaction conditions, no desired product was detected. When aliphatic 1*H*-tetrazole, such as 1-cyclohexyl-1*H*-tetrazole or 1-(*tert*-butyl)-1*H*-tetrazole was used in the reaction with benzaldehyde, only trace amount of product was observed.

In order to investigate the reaction mechanism, the related control experiments were carried out, shown in Scheme 2. When 1-phenyl-1*H*-tetrazole (**1a**) was treated with benzoic acid (**5a**) under the standard reaction conditions in the absence of TBHP, the reaction resulted in the formation of desired *N*-phenylbenzamide (**3aa**) in 85% yield (Scheme 2, eq. 1). Meanwhile, **1a** reacted with benzaldehyde (**2a**) in the absence of TBHP, only trace amount of **3aa** was detected (Scheme 2, eq. 2). Thus, the function of TBHP was probably as an oxidant to transform benzaldehyde **2a** into benzoic acid **5a**, which was also confirmed (Scheme 2, eq. 3). On the other hand, 1-phenyl-1*H*-tetrazole (**1a**) was transformed into *N*-phenylcyanamide **4a** in 48% yield without TBHP (Scheme 2, eq. 4). Simultaneously, treatment of **4a** with **2a** in the absence of TBHP, expected product **3aa** was isolated only in 25% yield (Scheme 2, eq. 5). When **4a** reacted with **2a** in the presence of TBHP, the desired product **3aa** was obtained 81% yield (Scheme 2, eq. 6). Importantly, the reaction of **4a** with **5a** in the absence of TBHP provided **3aa** in 89% yield (Scheme 2, eq. 7).

Based on our results and literature, a plausible mechanism for this reaction was proposed in Scheme 3. Firstly, 1-phenyl-1*H*-tetrazole (**1a**) was transformed into *N*-phenylcyanamide **4a** with losing of N₂.^{18d,18f} The obtained **4a** then reacted with benzoic acid (**5a**), generated from the oxidation of benzaldehyde (**2a**) by TBHP, to form the intermediate **6a**. Finally, **6a** underwent intramolecular elimination of cyano group by the reaction with H₂O in the system to give the desired product **3aa** and carbamic acid.



Scheme 2 The control experiments



Scheme 3 Proposal reaction mechanism

Conclusion

In summary, we have described an environmentally and friendly system for the synthesis of amides through the directly oxidative amidation of tetrazoles with aldehydes in the presence of TBHP as oxidant under transition-metal free conditions. A series of tetrazoles and aldehydes with different substituent afforded the desired products in good yields. The findings prove that the tetrazoles can serve as an amine equivalent in organic synthesis. It offers a novel, simple and mild method for the synthesis of amide derivatives. The detailed mechanistic study is currently underway.

Experimental Section

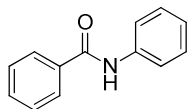
General remarks

All ^1H NMR and ^{13}C NMR spectra were recorded on a 400 MHz Bruker FT-NMR spectrometers (400 MHz and 100 MHz respectively). All chemical shifts are given as δ value (ppm) with reference to tetramethylsilane (TMS) as an internal standard. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, J , are reported in Hertz (Hz). The chemicals and solvents were purchased from commercial suppliers either from Aldrich, USA or Shanghai Chemical Company, China. All the tetrazole substrates were synthesized according to the reported procedure in the literature.¹⁹ Products were purified by flash chromatography on 100–200 mesh silica gels, SiO_2 .

Typical procedure for the reaction

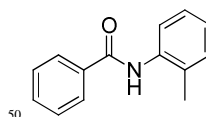
A sealable reaction flask equipped with a magnetic stirrer bar was charged with 1-phenyltetrazole (0.50 mmol), benzaldehyde (1.0 mmol), *tert*-butyl hydroperoxide (TBHP, 1.0 mmol) and DMF (2.0 mL). The mixture was stirred at 130 °C for 12 h [Caution: An Ace pressure tube is highly recommended to be employed for safety considerations]. After the reaction was finished, cooled to room temperature and diluted with ethyl acetate, washed with water and brine. And the organic phase was dried over MgSO_4 . After the solvent was removed under reduced pressure, the residue was purified by column chromatography on silica gel (eluant: petroleum ether with EtOAc in appropriate ratio) to afford the corresponding product.

Characterization data for all products



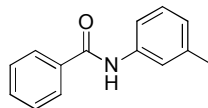
N-Phenylbenzamide (3aa)²⁰

80.8 mg, 82% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 8.00 (br, 1H), 7.87 (d, $J = 7.3$ Hz, 2H), 7.67–7.65 (m, 2H), 7.55–7.53 (m, 1H), 7.49–7.45 (m, 2H), 7.39–7.35 (m, 2H), 7.18–7.14 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.81, 137.94, 135.00, 131.76, 129.03, 128.72, 127.02, 124.54, 120.29.



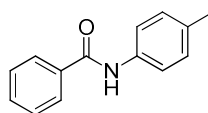
N-*o*-Tolylbenzamide (3ba)^{11j}

76.0 mg, 72% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 8.27 (br, s, 1H), 7.86–7.84 (m, 2H), 7.52–7.48 (m, 2H), 7.46–7.44 (m, 1H), 7.42–7.39 (m, 2H), 7.23–7.19 (m, 1H), 6.97–6.95 (m, 1H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.94, 138.76, 137.85, 134.92, 131.54, 128.67, 128.50, 127.01, 125.23, 121.05, 117.51, 21.32.



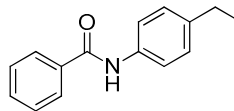
N-*m*-Tolylbenzamide (3ca)²¹

77.0 mg, 73% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 7.92 (br, s, 1H), 7.89–7.87 (m, 2H), 7.85–7.83 (m, 1H), 7.57–7.53 (m, 1H), 7.48–7.45 (m, 2H), 7.23–7.22 (m, 2H), 7.15–7.11 (m, 1H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.72, 135.69, 134.82, 131.65, 130.44, 129.87, 128.63, 127.02, 126.65, 125.40, 123.54, 17.71.



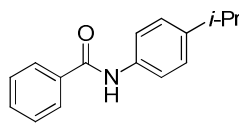
N-*p*-Tolylbenzamide (3da)²⁰

69.6 mg, 66% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 8.17 (br, s, 1H), 7.85 (d, $J = 7.4$ Hz, 2H), 7.55–7.53 (m, 2H), 7.51–7.49 (m, 1H), 7.43–7.40 (m, 2H), 7.14 (d, $J = 8.1$ Hz, 2H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.83, 135.38, 134.98, 134.07, 131.52, 129.40, 128.53, 127.01, 120.47, 20.80.



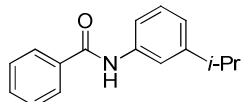
N-(4-Ethylphenyl)benzamide (3ea)²²

73.1 mg, 65% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 8.14 (br, s, 1H), 7.85 (d, $J = 7.4$ Hz, 2H), 7.56 (d, $J = 8.3$ Hz, 2H), 7.53–7.50 (m, 1H), 7.44–7.41 (m, 2H), 7.19–7.17 (m, 2H), 2.65 (q, $J = 7.6$ Hz, 2H), 1.25 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.86, 140.58, 135.53, 134.99, 131.56, 128.57, 128.26, 127.01, 120.56, 28.26, 15.55.



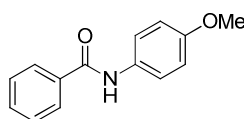
N-(4-*iso*-Propylphenyl)benzamide (3fa)²³

69.3 mg, 58% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 8.06 (br, s, 1H), 7.86 (d, $J = 7.4$ Hz, 2H), 7.57 (d, $J = 8.3$ Hz, 2H), 7.53–7.51 (m, 1H), 7.47–7.43 (m, 2H), 7.23–7.21 (m, 2H), 2.95–2.88 (m, 1H), 1.27 (d, $J = 6.9$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.87, 145.30, 135.56, 135.01, 131.63, 128.64, 127.01, 126.88, 120.54, 33.58, 23.96.



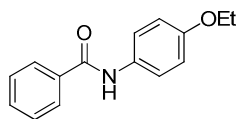
N-(3-iso-Propylphenyl)benzamide (3ga)

63.3 mg, 53% yield, Yellow solid, m. p. 107–108 °C. ^1H NMR (400 MHz, CDCl_3) δ : 8.15 (br, s, 1H), 7.89–7.87 (m, 2H), 7.55 (s, 1H), 7.53–7.50 (m, 2H), 7.46–7.43 (m, 2H), 7.30–7.26 (m, 1H), 7.05–7.03 (m, 1H), 2.94–2.87 (m, 1H), 1.27 (d, $J = 6.9$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.92, 149.94, 137.88, 134.99, 131.66, 128.88, 128.63, 127.01, 122.70, 118.50, 117.90, 34.07, 23.83. IR (KBr, cm^{-1}): 1650 ($\nu_{\text{C=O}}$). HRMS (ESI) $[\text{M}+\text{H}]^+$: Calcd. for $\text{C}_{16}\text{H}_{17}\text{NO}$: 240.1388. Found 240.1391.



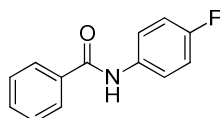
N-(4-Methoxyphenyl)benzamide (3ha)²⁰

69.2 mg, 61% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 7.92 (br, s, 1H), 7.87 (d, $J = 7.4$ Hz, 2H), 7.56–7.52 (m, 3H), 7.48–7.45 (m, 2H), 6.91–6.89 (m, 2H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.66, 156.63, 135.03, 131.63, 131.02, 128.68, 126.98, 122.15, 114.22, 55.48.



N-(4-Ethoxyphenyl)benzamide (3ia)²⁴

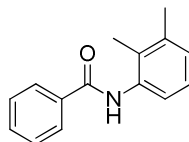
67.5 mg, 56% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 7.87–7.85 (m, 3H), 7.54–7.52 (m, 3H), 7.48–7.45 (m, 2H), 6.90–6.88 (m, 2H), 4.04 (q, $J = 6.8$ Hz, 2H), 1.42 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.63, 155.98, 135.05, 131.62, 130.88, 128.69, 126.96, 122.10, 114.84, 63.70, 14.81.



N-(4-Fluorophenyl)benzamide (3ja)^{11j}

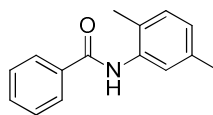
76.3 mg, 71% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 7.88–7.87 (m, 2H), 7.83 (br, s, 1H), 7.63–7.59 (m, 2H), 7.57–7.55 (m, 1H), 7.52–7.48 (m, 2H), 7.10–7.06 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.68, 159.58 (d, $J_{\text{C-F}} = 242.5$ Hz),

134.77, 133.91 (d, $J_{\text{C-F}} = 2.9$ Hz), 131.94, 128.83, 126.99, 122.09 (d, $J_{\text{C-F}} = 7.8$ Hz), 115.76 (d, $J_{\text{C-F}} = 22.4$ Hz).



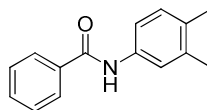
N-(2,3-Dimethylphenyl)benzamide (3ka)²⁵

67.5 mg, 60% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 7.91–7.89 (m, 2H), 7.77 (br, s, 1H), 7.59–7.55 (m, 2H), 7.51–7.48 (m, 2H), 7.17–7.13 (m, 1H), 7.08–7.06 (m, 1H), 2.34 (s, 3H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.89, 137.49, 135.33, 134.91, 131.72, 129.65, 128.73, 127.59, 127.09, 125.95, 122.24, 20.58, 13.85.



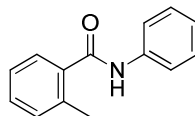
N-(2,5-Dimethylphenyl)benzamide (3la)²⁶

60.8 mg, 54% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 7.98 (br, s, 1H), 7.88 (d, $J = 7.4$ Hz, 2H), 7.62 (s, 1H), 7.56–7.52 (m, 1H), 7.46–7.43 (m, 2H), 7.11–7.09 (m, 1H), 6.95–6.93 (m, 1H), 2.32 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.71, 136.18, 135.37, 134.74, 131.50, 130.14, 128.50, 127.02, 126.97, 126.18, 124.27, 20.89, 17.19.



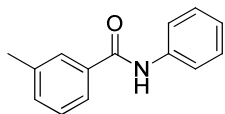
N-(3,4-Dimethylphenyl)benzamide (3ma)²⁷

77.6 mg, 69% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 8.23 (br, s, 1H), 7.86 (d, $J = 7.4$ Hz, 2H), 7.51–7.48 (m, 1H), 7.45 (s, 1H), 7.41–7.38 (m, 3H), 7.08–7.06 (m, 1H), 2.24 (s, 3H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.83, 137.02, 135.63, 134.98, 132.71, 131.41, 129.81, 128.44, 127.00, 121.83, 118.03, 19.68, 19.06.

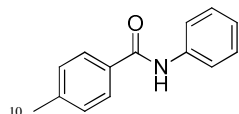


2-Methyl-N-phenylbenzamide (3ab)^{11j}

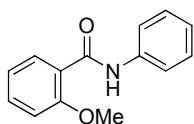
61.2 mg, 58% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 7.78 (br, s, 1H), 7.63–7.61 (m, 2H), 7.44–7.42 (m, 1H), 7.37–7.33 (m, 3H), 7.25–7.22 (m, 2H), 7.17–7.14 (m, 1H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 168.15, 137.99, 136.37, 136.26, 131.10, 130.11, 128.96, 126.59, 125.75, 124.41, 119.91, 19.69.

**3-Methyl-N-phenylbenzamide (3ac)**^{11j}69.6 mg, 66% yield, Colorless solid. ¹H NMR (400 MHz, CDCl₃)

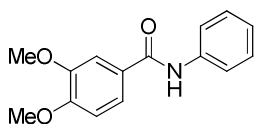
δ: 7.93 (br, s, 1H), 7.69 (s, 1H), 7.67–7.65 (m, 3H), 7.39–7.35 (m, 4H), 7.17–7.14 (m, 1H), 2.42 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 166.34, 138.24, 138.02, 134.73, 132.22, 128.72, 128.24, 127.79, 124.23, 124.01, 120.46, 21.09.

**4-Methyl-N-phenylbenzamide (3ad)**^{11j}85.5 mg, 81% yield, Colorless solid. ¹H NMR (400 MHz, CDCl₃)

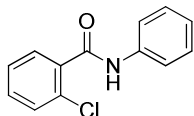
δ: 8.18 (br, s, 1H), 7.76 (d, *J* = 7.9 Hz, 2H), 7.66 (d, *J* = 7.3 Hz, 2H), 7.35–7.32 (m, 2H), 7.23–7.21 (m, 2H), 7.15–7.12 (m, 1H), 2.40 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 165.88, 142.13, 138.07, 132.04, 129.23, 128.88, 127.05, 124.29, 120.33, 21.35.

**2-Methoxy-N-phenylbenzamide (3ae)**²⁸54.5 mg, 48% yield, Colorless solid. ¹H NMR (400 MHz, CDCl₃)

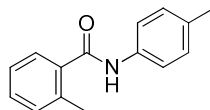
δ: 9.81 (br, s, 1H), 8.31–8.29 (m, 1H), 7.71–7.69 (m, 2H), 7.51–7.47 (m, 1H), 7.39–7.35 (m, 2H), 7.15–7.12 (m, 2H), 7.04–7.02 (m, 1H), 4.04 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 163.17, 157.15, 138.35, 133.16, 132.43, 128.90, 124.07, 121.76, 121.59, 120.38, 111.50, 56.16.

**3,4-Dimethoxy-N-phenylbenzamide (3af)**²⁹65.5 mg, 51% yield, Colorless solid. ¹H NMR (400 MHz, CDCl₃)

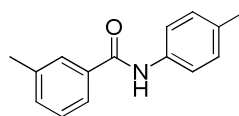
δ: 8.16 (br, s, 1H), 7.65 (d, *J* = 7.9 Hz, 2H), 7.47 (s, 1H), 7.42–7.40 (m, 1H), 7.35–7.32 (m, 2H), 7.14–7.11 (m, 1H), 6.83–6.81 (m, 1H), 3.89 (s, 3H), 3.86 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 165.43, 151.97, 149.06, 138.12, 128.93, 127.46, 124.29, 120.25, 119.58, 110.73, 110.28, 55.93, 55.89.

**2-Chloro-N-phenylbenzamide (3ag)**³⁰50.8 mg, 44% yield, Colorless solid. ¹H NMR (400 MHz, CDCl₃)

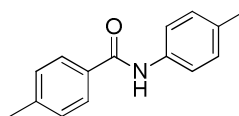
δ: 8.10 (br, s, 1H), 7.69–7.63 (m, 3H), 7.43–7.33 (m, 5H), 7.19–7.15 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ: 164.57, 137.55, 135.20, 131.52, 130.58, 130.25, 130.08, 129.01, 127.14, 124.75, 120.11.

**2-Methyl-N-p-tolylbenzamide (3db)**³¹66.4 mg, 59% yield, Colorless solid. ¹H NMR (400 MHz, CDCl₃)

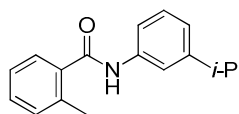
δ: 7.64 (br, s, 1H), 7.50 (d, *J* = 7.7 Hz, 2H), 7.45–7.43 (m, 1H), 7.37–7.33 (m, 1H), 7.26–7.22 (m, 2H), 7.17–7.16 (m, 2H), 2.48 (s, 3H), 2.35 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 167.99, 136.49, 136.28, 135.41, 134.08, 131.10, 130.06, 129.47, 126.57, 125.75, 119.93, 20.84, 19.73.

**3-Methyl-N-p-tolylbenzamide (3dc)**³¹69.8 mg, 62% yield, Colorless solid. ¹H NMR (400 MHz, CDCl₃)

δ: 8.17 (br, s, 1H), 7.66 (s, 1H), 7.63–7.62 (m, 1H), 7.54 (d, *J* = 7.6 Hz, 2H), 7.31–7.27 (m, 2H), 7.14 (d, *J* = 7.6 Hz, 2H), 2.37 (s, 3H), 2.34 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 166.00, 138.38, 135.43, 134.93, 133.93, 132.25, 129.36, 128.37, 127.75, 123.94, 120.38, 21.22, 20.80.

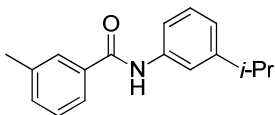
**4-Methyl-N-p-tolylbenzamide (3dd)**²⁰78.8 mg, 70% yield, Colorless solid. ¹H NMR (400 MHz, CDCl₃)

δ: 8.14 (br, s, 1H), 7.75 (d, *J* = 7.9 Hz, 2H), 7.53 (d, *J* = 8.2 Hz, 2H), 7.21 (d, *J* = 7.9 Hz, 2H), 7.13 (d, *J* = 8.1 Hz, 2H), 2.40 (s, 3H), 2.34 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 165.74, 141.96, 135.48, 133.86, 132.07, 129.35, 129.17, 127.01, 120.38, 21.35, 20.80.

**N-(3-iso-Propylphenyl)-2-methylbenzamide (3g)**73.4 mg, 58% yield, Yellow liquid. ¹H NMR (400 MHz, CDCl₃) δ:

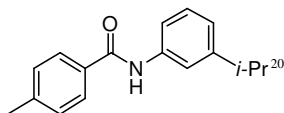
7.91 (br, s, 1H), 7.52–7.49 (m, 2H), 7.42–7.40 (m, 1H), 7.36–7.32 (m, 1H), 7.30–7.27 (m, 1H), 7.24–7.18 (m, 2H), 7.06–7.04 (m, 1H), 2.95–2.88 (m, 1H), 2.47 (s, 3H), 1.29 (d, *J* =

6.8 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 168.09, 149.85, 138.01, 136.45, 136.17, 130.99, 129.96, 128.82, 126.59, 125.65, 122.42, 118.03, 117.41, 34.03, 23.81, 19.65. IR (KBr, cm^{-1}): 1654 ($\nu_{\text{C=O}}$). HRMS (ESI) $[\text{M}+\text{H}]^+$: Calcd. for $\text{C}_{17}\text{H}_{19}\text{NO}$: 254.1545. Found 254.1544.



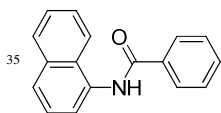
N-(3-iso-Propylphenyl)-3-methylbenzamide (3gc)

82.2 mg, 65% yield, Yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ : 8.27 (br, s, 1H), 7.70 (s, 1H), 7.67–7.65 (m, 1H), 7.57 (s, 1H), 7.54–7.52 (m, 1H), 7.31–7.29 (m, 2H), 7.27–7.25 (m, 1H), 7.04–7.02 (m, 1H), 2.95–2.86 (m, 1H), 2.37 (s, 3H), 1.26 (d, $J = 6.9$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 166.04, 149.79, 138.39, 138.00, 134.95, 132.29, 128.77, 128.39, 127.77, 123.97, 122.46, 118.41, 117.81, 34.02, 23.80, 21.20. IR (KBr, cm^{-1}): 1649 ($\nu_{\text{C=O}}$). HRMS (ESI) $[\text{M}+\text{H}]^+$: Calcd. for $\text{C}_{17}\text{H}_{19}\text{NO}$: 254.1545. Found 254.1543.



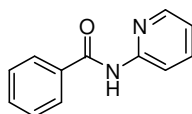
N-(3-iso-Propylphenyl)-4-methylbenzamide (3gd)

88.5 mg, 70% yield, Yellow liquid. ^1H NMR (400 MHz, CDCl_3) δ : 8.24 (br, s, 1H), 7.78 (d, $J = 8.0$ Hz, 2H), 7.57 (s, 1H), 7.53–7.51 (m, 1H), 7.26 (t, $J = 7.8$ Hz, 1H), 7.21 (d, $J = 7.7$ Hz, 2H), 7.03–7.01 (m, 1H), 2.94–2.84 (m, 1H), 2.40 (s, 3H), 1.26 (d, $J = 6.9$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.80, 149.79, 142.01, 138.06, 132.11, 129.18, 128.76, 127.03, 122.39, 118.41, 117.81, 34.03, 23.80, 21.33. IR (KBr, cm^{-1}): 1649 ($\nu_{\text{C=O}}$). HRMS (ESI) $[\text{M}+\text{H}]^+$: Calcd. for $\text{C}_{17}\text{H}_{19}\text{NO}$: 254.1545. Found 254.1541.



N-(Naphthalen-1-yl)benzamide (3na)²¹

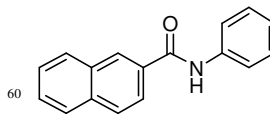
58.0 mg, 47% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 8.40 (br, s, 1H), 7.96–7.94 (m, 2H), 7.89–7.87 (m, 3H), 7.74–7.72 (m, 1H), 7.58–7.55 (m, 1H), 7.52–7.45 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ : 166.37, 134.64, 134.05, 132.36, 131.80, 128.68, 128.64, 127.62, 127.17, 126.25, 126.11, 125.94, 125.61, 121.52, 120.91.



N-(Pyridin-2-yl)benzamide (3oa)³²

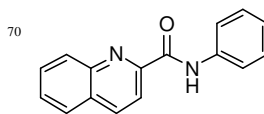
50.5 mg, 51% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 9.53 (br, s, 1H), 8.42–8.40 (m, 1H), 8.03–8.02 (m, 1H),

7.92–7.91 (m, 2H), 7.72–7.68 (m, 1H), 7.53–7.50 (m, 1H), 7.44–7.43 (m, 2H), 6.97–6.96 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 166.11, 151.77, 147.61, 138.32, 134.37, 131.69, 128.56, 127.30, 119.63, 114.35.



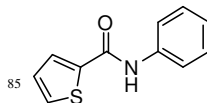
N-Phenyl-2-naphthamide (3ah)³³

67.9 mg, 55% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 8.37 (s, 1H), 8.12 (br, s, 1H), 7.93–7.88 (m, 4H), 7.71 (d, $J = 7.9$ Hz, 2H), 7.61–7.54 (m, 2H), 7.42–7.38 (m, 2H), 7.20–7.16 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 165.92, 137.95, 134.84, 132.58, 132.13, 129.10, 128.94, 128.72, 127.88, 127.78, 127.53, 126.91, 124.62, 123.52, 120.32.



N-Phenylquinoline-2-carboxamide (3ai)³⁴

60.8 mg, 49% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 10.24 (br, s, 1H), 8.43–8.36 (m, 2H), 8.21–8.19 (m, 1H), 7.93–7.91 (m, 1H), 7.88–7.86 (m, 2H), 7.84–7.80 (m, 1H), 7.68–7.64 (m, 1H), 7.45–7.42 (m, 2H), 7.21–7.17 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 162.13, 149.65, 146.27, 137.81, 137.78, 130.28, 129.64, 129.40, 129.08, 128.12, 127.78, 124.31, 119.74, 118.72.



N-Phenylthiophene-2-carboxamide (3aj)²⁹

38.6 mg, 38% yield, Colorless solid. ^1H NMR (400 MHz, CDCl_3) δ : 7.91 (br, s, 1H), 7.66–7.65 (m, 1H), 7.63–7.61 (m, 2H), 7.54–7.53 (m, 1H), 7.37–7.33 (m, 2H), 7.16–7.13 (m, 1H), 7.10 (t, $J = 4.3$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 160.03, 139.28, 137.58, 130.72, 129.03, 128.47, 127.77, 124.58, 120.31.

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Notes and reference

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