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## HYDROGEN BOND ORGANOCATALYSIS OF BENZOTRIAZOLE IN TRANSAMIDATION OF CARBOXAMIDES WITH AMINES

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**Abstract** – A new method of transamidation of carboxamides with amines catalyzed by benzotriazole has been developed.<sup>†</sup>

### INTRODUCTION

Amide bonds are ubiquitous in living systems and play a key role in bioorganic and medicinal chemistry. Carboxamide group was found to appear in more than 25% of known drugs.<sup>1</sup> Amide formation is thus one of the most fundamental reactions in organic synthesis.<sup>2</sup> The most common strategy for amide bond formation is the reaction between amines and carboxylic acids in the presence of stoichiometric amounts of activating reagents. For this approach, the first step is the activation of the acid (acid chlorides, acylimidazoles, acylazides, anhydrides, and active esters), followed by aminolysis. Much effort has been made to develop a catalytic direct amidation.<sup>3</sup>

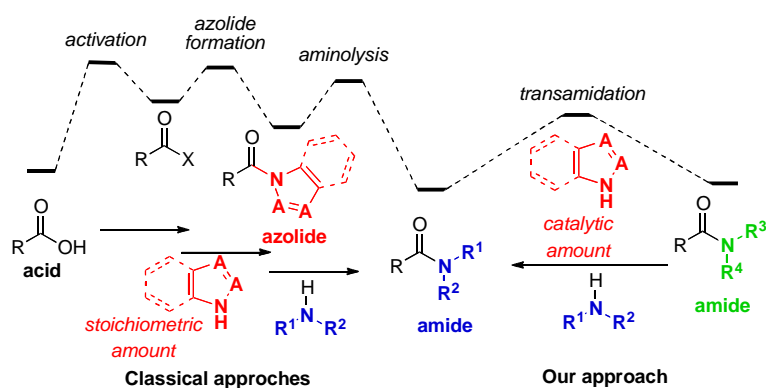
Alternatively, transamidation reaction between a carboxamide and an amine is an attractive tool in synthetic organic chemistry. However, uncatalyzed transamidation required in general drastic heating conditions.<sup>4</sup> Different methods utilizing activating reagents or catalysts have been developed with the objective to decrease the reaction temperature.<sup>5</sup> Despite their wide scope, these protocols required at least one of these conditions: (i) energetically favorable systems (ring-opening of four-membered rings,<sup>5d,e</sup> (ii) intramolecular assistance,<sup>5f</sup> or both factors); (iii) use of moisture-sensitive and/or activation reagents (up to 2–3 equivalents; borate esters,<sup>5a</sup> dialkylformamide dialkyl acetals,<sup>5b</sup> AlCl<sub>3</sub>,<sup>5g</sup> AlMe<sub>3</sub>,<sup>5c</sup> HCl<sup>5i</sup>). Examples of transamidation from the Stahl's group provided an elegant possibility of preparing amides under mild

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<sup>†</sup> This paper is dedicated to Prof. Victor Snieckus on the occasion of his 77<sup>th</sup> birthday.

conditions.<sup>6</sup> However, the reaction requires expensive catalyst and can also result in mixtures of amides. Recently, new copper-<sup>7</sup> and cerium-,<sup>8</sup> and zirconium-catalyzed<sup>9</sup> transamidations have been developed for carboxamides,<sup>7-9</sup> and ureas.<sup>7</sup> For metal-free catalyzed methods of transamidation,  $\text{NH}_2\text{OH}\cdot\text{HCl}$  (up to 50 mol%),<sup>10</sup> and  $\text{NH}_3\cdot\text{HI}$  (1 equiv)<sup>11</sup> were reported to be catalytically active. In reality, because the free bases  $\text{NH}_2\text{OH}$  ( $pK_{\text{aNH}_3\text{OH}^+} = 5.97$ ) and  $\text{NH}_3$  ( $pK_{\text{aNH}_4^+} = 9.21$ ) are less basic than most alkyl amines (for example,  $pK_{\text{aBnNH}_3^+} = 9.34$ ,  $pK_{\text{aCyclohexylNH}_3^+} = 10.64$ )<sup>12</sup> and because ammonia is more volatile than any amine, the corresponding salts  $\text{NH}_2\text{OH}\cdot\text{HCl}$  and  $\text{NH}_3\cdot\text{HI}$  would transfer their acid to alkyl amines to provide aliphatic ammonium, which could probably be the principle catalytic species in these two examples.<sup>5i</sup> In the case of  $\text{NH}_3\cdot\text{HI}$ , high reactivities with *N*-alkylamines were observed at the expense of using excess of ethylenediamine - a strong and chelating base - under microwave activation. Very recently,  $\text{PhI}(\text{OAc})_2$ ,<sup>13</sup> glycine<sup>14</sup> and dioxane<sup>15</sup> were shown to be capable of catalyzing the transamidation reaction.

We recently reported a highly convenient access to amides by solvent-free boric acid-catalyzed transamidation of carboxamides with amines.<sup>16</sup> In conjunction with this work, we became interested in achieving such transformation using a new class of robust, tunable catalysts under practical and mild conditions. In this regard, azoles (imidazoles,<sup>17</sup> pyrazoles, triazoles,<sup>18</sup> benzimidazoles, benzotriazoles,<sup>19</sup> etc.) would be especially attractive candidates. The electronic nature of the azoles is dictated by the number and position of the nitrogen atoms and therefore could determine the effectiveness of the azole catalyst (Scheme 1). Classically, their *N*-acyl derivatives – azolides – have proved to be effective acylating agents for amines.<sup>20</sup> However, it should be kept in mind that this method encounters two intrinsic drawbacks: (i) the formation of azolides from the corresponding acids requires azoles in stoichiometric amounts and an additional step of activation; and (ii) the subsequent aminolysis of azolides will release inevitably the parent azoles as by-products.



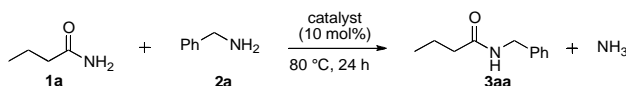
**Scheme 1.** Amide formation involving azoles

## RESULTS AND DISCUSSION

To identify efficient transamidation catalysts, we screened several commercially available azoles (10

mol%) for their ability to catalyze the reaction of butyramide **1a** with benzylamine **2a** under solvent-free conditions (Table 1).

**Table 1.** Reaction conditions screening<sup>a</sup>



Entry	Catalyst	Conversion (%) <sup>b</sup>
1	-	<5
2	imidazole	<5
3	<i>N</i> -methylimidazole	<5
4	benzimidazole	<5
5	pyrazole	12
6	indazole	10
7	1,2,4-triazole	34
8	1,2,3-triazole	35
9	benzotriazole	51
10	1-methylbenzotriazole	19
11	2-methylbenzotriazole	21

<sup>a</sup> Conditions: **1a** (5 mmol), **2a** (5 mmol), catalyst (0.5 mmol) under Ar for 24 h. <sup>b</sup> Determined by <sup>1</sup>H NMR.

At 80 °C for 24 h, the uncatalyzed transamidation did not occur (Entry 1). Unsubstituted imidazole (Entry 2) and its derivatives (Entries 3-4) showed only negligible effect. A slight catalytic effect was observed with pyrazole and indazole (Entries 5-6).



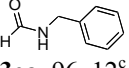


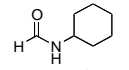

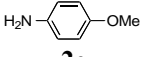
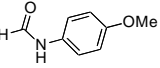

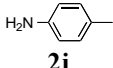
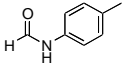


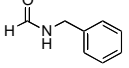

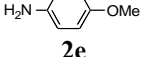
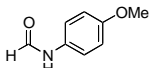

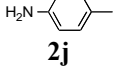
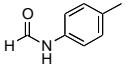


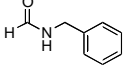

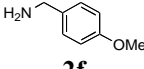
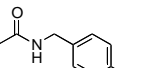


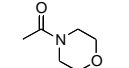


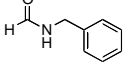

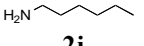
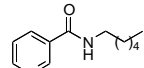


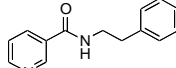

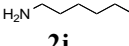
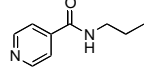


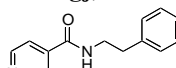
We next turned our attention to the use of triazole derivatives. To our delight, simple triazoles showed enhanced catalytic effect (Entries 7-8) compared to pyrazoles. We theorized that the presence of at least two nitrogen atoms bonded together might be responsible for this catalytic effect. The most remarkable conversion was obtained with benzotriazole (Entry 9). Finally, two *N*-substituted benzotriazoles were tested and displayed considerably lower efficiency (Entries 10-11).

The scope of the benzotriazole catalyzed transamidation was examined by reaction of a number of structurally diverse amides and amines using 10% catalyst (Table 2) on a preparative scale. The reactions of *n*-butyramide **1a** and acetamide **1b** proceeded cleanly with aliphatic primary and secondary amines (**2a-d, f** and **g**) bearing various substituents at moderate temperatures. The desirable carboxamides were obtained in high yields (Entries 1-4, 6-8). While the reaction of **1a** with **2c** occurred without incidence, the transamidation of **1a** with **2d** provided small quantity of ring closing product, probably due to higher temperature required in the latter case. With low nucleophilic aromatic amine **2e**, the transamidation required higher temperature to afford the product in useful yields (Entries 5 and 9). Aromatic substrates such as benzamide **1c** and *p*-toluamide **1d** also reacted well although heating at higher temperature is required (Entries 10-15). Interestingly, formamide **1e** displayed a very high reactivity under the present conditions. With benzylamine **2a**, the reaction achieved full conversion even at room temperature (Entry 16). Reaction with hindered amine such as cyclohexylamine required heating only at 50 °C (Entry 17). At

100 °C, **1e** reacts cleanly with aromatic amines (Entries 18-19). These promising results with **1e** suggest that **1e** could be an attracting alternative formylation reagent using our benzotriazole catalyst.<sup>17</sup>

**Table 2.** Transamidation scope and limitations<sup>a</sup>

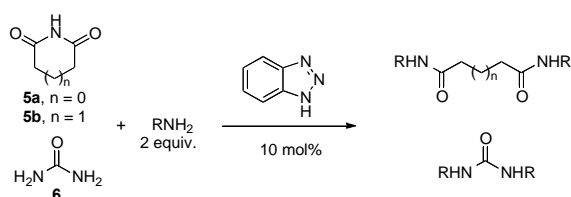
Entry	Amide	Amine	Temp (°C)	Product, Yield (%)	
1			110	 <b>3aa</b> , 95	
2			120	 <b>3ab</b> , 93	
3			120	 <b>3ac</b> , 97	
4			150	 <b>3ad</b> , 6	 <b>3ad'</b> , 15 <sup>b</sup>
5			150	 <b>3ae</b> , 42	
6			160	 <b>3ae</b> , 70	
7			120	 <b>3bf</b> , 85	
8			130	 <b>3bg</b> , 83	
9			130	 <b>3be</b> , 50	
10			150	 <b>3ch</b> , 80	
11			140	 <b>3ca</b> , 83	
12			150	 <b>3ce</b> , 40 <sup>b</sup>	
13			120	 <b>3db</b> , 66	
14			120	 <b>3dc</b> , 55	
15			150	 <b>3dd</b> , 44	

16			rt	
	<b>1e</b>	<b>2a</b>		<b>3ea</b> , 96, 12 <sup>c</sup>
17			30 50	
	<b>1e</b>	<b>2i</b>		<b>3ei</b> , 33, <sup>b</sup> 3 <sup>c</sup> <b>3ei</b> , 89
18			80 100	
	<b>1e</b>	<b>2e</b>		<b>3ee</b> , 44, <sup>b</sup> 10 <sup>c</sup> <b>3ee</b> , 90
19			100	
	<b>1e</b>	<b>2j</b>		<b>3ej</b> , 82
20			80	
	<b>1e'</b>	<b>2a</b>		<b>3ea</b> , 85
21			150	
	<b>1e'</b>	<b>2e</b>		<b>3ee</b> , 50
22			150	
	<b>1e'</b>	<b>2j</b>		<b>3ej</b> , 60
23			150	
	<b>1e''</b>	<b>2a</b>		<b>3ea</b> , 87
24			130	
	<b>1b'</b>	<b>2f</b>		<b>3bf</b> , 50
25			150	
	<b>1b'</b>	<b>2g</b>		<b>3bg</b> , 40 <sup>b</sup>
26			150	
	<b>1b''</b>	<b>2a</b>		<b>3ba</b> , 35
27			140	
	<b>1f</b>	<b>2j</b>		<b>3fj</b> , 80
28			150	
	<b>1f</b>	<b>2h</b>		<b>3fh</b> , 96
29			150	
	<b>1g</b>	<b>2j</b>		<b>3gj</b> , 80
30			150	
	<b>1g</b>	<b>2h</b>		<b>3gh</b> , 96

<sup>a</sup> Reaction conditions: amide (5 mmol), amine (5 mmol), benzotriazole (0.5 mmol) under Ar for 24 h unless otherwise noted. <sup>b</sup> Conversion determined by <sup>1</sup>H NMR. <sup>c</sup> Conversion at the same temperature of the background reaction.

The transamidation reactivity of *N*-monosubstituted (**1f'**,**1h'**) and *N,N*-disubstituted amides (**1f''**,**1h''**) were next evaluated with amines (Entries 20-26). As a general trend, the reactivity of amides is reduced significantly with their *N*-substitution. Finally, our method can be applied successfully to heterocyclic amides such as nicotinamide **1f** and isonicotinamide **1g** which could provide new rapid access to biologically important *N*-substituted nicotinamides starting from readily available unsubstituted nicotinamide.

**Table 3.** Transamidation of Imides and Urea<sup>a</sup>



Entry	Imide/Urea	Amine	Temp (°C)	Product, Yield (%)
1	 <b>1h</b>	 <b>2a</b>	130	 <b>3ha</b> , 90
2	 <b>1h</b>	 <b>2i</b>	140	 <b>3hi</b> , 92
3	 <b>1i</b>	 <b>2a</b>	130	 <b>3ia</b> , 97
4 <sup>c</sup>	 <b>1j</b>	 <b>2a</b>	80	 <b>3ja</b> , 0 <b>3ja'</b> , 0
5	 <b>1j</b>	 <b>2a</b>	80	 <b>3ja</b> , 25
6	 <b>1j</b>	 <b>2a</b>	120	 <b>3ja'</b> , 94
7	 <b>1j</b>	 <b>2h</b>	120	 <b>3jh</b> , 80
8	 <b>1j</b>	 <b>2e</b>	120	 <b>3je</b> , 54
9 <sup>b</sup>	 <b>1j</b>	 <b>2k</b>	120	 <b>3jk</b> , 97
10 <sup>b</sup>	 <b>1j</b>	 <b>2l</b>	140	 <b>3jl</b> , 75

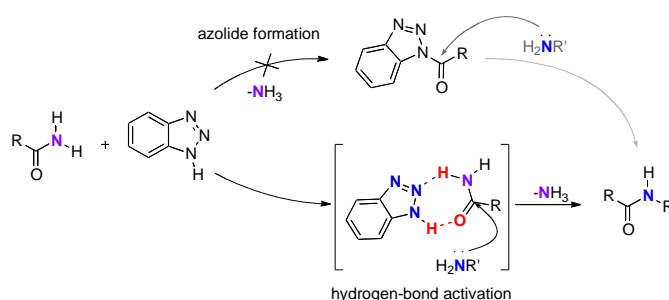
<sup>a</sup> Conditions: imide/urea (5 mmol), amine (10 mmol), benzotriazole (0.5 mmol) under Ar for 24 h unless otherwise noted. <sup>b</sup> Amine (5 mmol) was used. <sup>c</sup> Background reaction.

To fulfil our study, we turn our attention to the application of the method to imides and urea (Table 3). Succinimide **1h** and glutarimide **1i** were subjected to standard reaction conditions with 2 equiv of amines (Entries 1-3). Complete conversion of the starting materials was observed. Unsubstituted urea **1j** can react with different monofunctional aliphatic and aromatic amines (Entries 5-8). Unsurprisingly, the reaction of urea **1j** with diamines **2k** and **2l** leads to the corresponding cyclic **3jk** and **3jl** ureas in high yields (Entries 9 and 10).

One possible supposition for this catalytic effect is that benzotriazole attacks the starting amide, generating an activated azolide for the second step of aminolysis (Scheme 2). To test this hypothesis, an equimolar mixture of benzotriazole and acetamide was heated at 150 °C for 24 h. Although the formation of gaseous ammoniac could be a thermodynamically favorable factor for this hypothesized reaction, both starting materials were recovered unchanged, excluding this reaction pathway.

We next focused our attention on another mode of activation by formation of cooperative hydrogen bonds as a driving force to explain benzotriazole-catalyzed transamidation. It is possible that benzotriazole activates effectively the starting amide by forming two hydrogen bonds (Scheme 2).<sup>21</sup> This intermolecular association by hydrogen bond would allow an easy aminolysis by the primary or secondary amine with loss of ammonia.

This hypothesis is supported by the fact that when imidazole derivatives (Table 1, Entries 2-4) or *N*-methylated benzotriazoles (Table 1, Entries 10-11) were used, the formation of only one hydrogen bond is possible between the catalyst and the starting amide. Consequently, we consider that the second nitrogen atom in benzotriazole is required to fix the amide and the catalyst closely, resulting in an efficient activation for subsequent aminolysis.



**Scheme 2.** Proposed reaction mechanism

In summary, we have identified benzotriazole as an efficient organocatalyst for transamidation reaction. This catalyst can be applied efficiently in the preparation of diversely substituted amides bearing different functional groups under mild conditions. The presence of two contiguous nitrogen atoms in the catalyst is of crucial importance. Overall, this method represents an operationally simple and an easily scalable. Further studies on transamidation reaction are currently investigated in our group and will be reported in due course.

## EXPERIMENTAL

**General procedure for transamidation reaction.** A mixture of amide (5 mmol), amine (5 mmol) and benzotriazole (60 mg, 0.5 mmol) was stirred in a tube at indicated temperature for indicated reaction time (See Tables 1-5). After being cooled to room temperature, the crude reaction mixture was purified by recrystallization or silica gel column chromatography (dissolved in dichloromethane, eluted with dichloromethane and ethyl acetate).

***N*-Benzylbutyramide (3aa).**<sup>9</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 98:2) afforded the product as a white powder. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.36-7.27 (m, 5H), 5.85 (broad s, 1H), 4.46 (d, *J* = 5.8 Hz, 2H), 2.20 (t, *J* = 7.3 Hz, 2H), 1.70 (sextuplet, *J* = 7.3 Hz, 2H), 0.97 (t, *J* = 7.3 Hz, 3H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 173.1, 138.7, 128.7, 127.8, 127.5, 43.6, 38.7, 19.3, 13.9.

***N*-(4-Hydroxybutyl)butyramide (3ab).** Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 1:1) afforded the product as brown oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 6.33 (br. s, 1H), 3.84 (br. s, 1H), 3.67 (m, 2H), 3.28 (m, 2H), 2.17 (t, *J* = 8.6 Hz, 2H), 1.64 (m, 6H), 0.94 (t, *J* = 8.6 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 174.0, 62.3, 39.7, 39.1, 29.8, 26.4, 19.2, 13.5. HRMS (ESI+) *m/z* [M+H]<sup>+</sup> calcd for C<sub>8</sub>H<sub>18</sub>NO<sub>2</sub> 160.1338, found 160.1330.

***N*-(2-Hydroxyethyl)butyramide (3ac).**<sup>22</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 1:1) afforded the product as brown oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 6.72 (br. s, 1H), 4.11 (br. s, 1H), 3.71 (t, *J* = 5.7 Hz, 2H), 3.41 (m, 2H), 2.21 (t, *J* = 7.3 Hz, 2H), 1.67 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 174.5, 62.4, 42.6, 38.7, 19.5, 13.7.

***N*-(2-Hydroxy-1-phenylethyl)butyramide (3ad).**<sup>23</sup> Purification of the crude mixture by kugelrohr removal of volatiles (120 °C, 0.1 mmHg) followed by column chromatography (heptane:AcOEt 1:1) afforded the product as brown oil which crystallized slowly on standing. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) (two rotamers 9:1) δ 7.93 (d, *J* = 8.1 Hz, 1H), 7.15-7.00 (m, 5H), 6.57 (br. s, 1H), 5.49 (m, 1H), 4.92 (m, 2H), 2.53 (br. s, 1H), 2.13 (t, *J* = 7.7 Hz, 2H), 1.56 (m, 2H), 0.81 (t, *J* = 7.3 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) (two rotamers) δ 172.9, 138.0, 129.1, 128.3, 127.7, 126.6, 124.3, 119.8, 109.4, 53.2, 52.4, 38.5, 19.0, 13.8.

***N*-(4-Methoxyphenyl)butyramide (3ae).**<sup>9</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 95:5) afforded the product as dark solid. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.60 (br. s, 1H), 7.44 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 3.80 (s, 3H), 2.32 (t, *J* = 7.7 Hz, 2H), 1.76 (m, 2H), 1.01 (t, *J* = 7.3 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 171.5, 156.3, 131.2, 121.9, 114.1, 55.5, 39.4, 19.1, 13.7.

***N*-(4-Methoxyphenyl)acetamide (3be).**<sup>24</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 9:1) afforded the product as brown powder. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.41 (m, 3H),



6.86 (d,  $J = 8.3$  Hz), 2H; 3.80 (s, 3H), 2.16 (s, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ),  $\delta$  168.4, 156.4, 130.9, 122.1, 114.3, 55.8, 24.4.

***N*-(4-Methoxybenzyl)acetamide (3bf).**<sup>9</sup> Purification of the crude mixture by column chromatography ( $\text{CH}_2\text{Cl}_2$ :AcOEt 80:20) afforded the product as a white powder.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ),  $\delta$  7.24 (d,  $J = 8.8$  Hz, 2H), 6.88 (d,  $J = 8.8$  Hz, 2H), 5.86 (br. s, 1H), 4.37 (d,  $J = 5.8$  Hz, 2H), 3.81 (s, 3H), 2.03 (s, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ),  $\delta$  169.9, 159.2, 130.5, 129.4, 114.2, 55.8, 43.6, 23.3.

**1-Morpholinoethanone (3bg).**<sup>9</sup> Purification of the crude mixture by column chromatography ( $\text{CH}_2\text{Cl}_2$ :AcOEt 8:2) afforded the product as colorless oil.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ),  $\delta$  3.65 (m, 4H), 3.60 (m, 2H), 2.08 (s, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ),  $\delta$  169.2, 66.8, 66.6, 46.8, 41.7, 21.0.

***N*-Benzylbenzamide (3ca).**<sup>25</sup> Purification of the crude mixture by column chromatography ( $\text{CH}_2\text{Cl}_2$ :AcOEt 8:2) afforded the product as colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ),  $\delta$  7.76 (d,  $J = 8.8$  Hz, 2H), 7.48-7.24 (m, 8H), 6.52 (broad s, 1H), 4.63 (d,  $J = 5.8$  Hz, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ),  $\delta$  167.7, 138.4, 134.5, 131.6, 128.8, 128.6, 127.9, 127.6, 127.2, 44.2.

***N*-Phenethylbenzamide (3ch).**<sup>26</sup> Purification of the crude mixture by column chromatography ( $\text{CH}_2\text{Cl}_2$ :AcOEt 9:1) afforded the product as white crystals.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.78-7.22 (m, 10H), 6.27 (br. s, 1H), 3.77 (dd,  $J = 13.3, 7.6$  Hz, 2H), 2.98 (t,  $J = 13.3$  Hz, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  167.6, 139.0, 134.7, 131.4, 128.9, 128.8, 128.6, 126.8, 126.6, 41.1, 35.7.

***N*-(4-Hydroxybutyl)-4-methylbenzamide (3db).** Purification of the crude mixture by kugelrohr removal of volatiles (120 °C, 0.1 mmHg) followed by column chromatography (heptane:AcOEt 1:1) afforded the product as brown oil.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 (d,  $J = 8.0$  Hz, 2H), 7.20 (d,  $J = 8.0$  Hz, 2H), 6.83 (br. s, 1H), 3.70 (t,  $J = 6.2$  Hz, 2H), 3.49 (dd,  $J = 13.0, 6.2$  Hz, 2H), 2.40 (s, 3H), 1.69 (m, 4H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  167.8, 141.8, 129.4, 127.1, 125.5, 62.3, 39.8, 30.1, 26.4, 21.6. HRMS (ESI+)  $m/z$   $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{12}\text{H}_{18}\text{NO}_2$  208.1338, found 208.1345.

***N*-(2-Hydroxyethyl)-4-methylbenzamide (3dc).**<sup>27</sup> Purification of the crude mixture by kugelrohr removal of volatiles (120 °C, 0.1 mmHg) followed by column chromatography (heptane:AcOEt 1:1) afforded the product as brown oil.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 (d,  $J = 8.5$  Hz, 2H), 7.21 (d,  $J = 8.5$  Hz, 2H), 6.99 (br. s, 1H), 3.82 (m, 2H), 3.61 (m, 2H), 2.39 (s, 3H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  168.8, 142.2, 131.3, 129.3, 127.3, 62.1, 43.1, 21.7.

***N*-Benzylformamide (3ea).**<sup>16</sup> Purification of the crude mixture by column chromatography ( $\text{CH}_2\text{Cl}_2$ :AcOEt 98:2) afforded the product as a white powder.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ), two rotamers.  $\delta$  8.24 [s, d ( $J = 12.0$  Hz), 1H], 7.50-7.20 (m, 5H), 6.38 (m, 1H), [4.48 (d,  $J = 6.0$  Hz), 4.42 (d,  $J = 6.0$  Hz), 2H].  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ), two rotamers.  $\delta$  165.0, 161.3, 137.6, 128.9, 128.8, 128.0, 127.8, 127.7, 127.0, 45.6, 42.3.

***N*-(4-Methoxyphenyl)formamide (3ee).**<sup>16</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 95:5) afforded the product as brown powder. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>), two rotamers.  $\delta$  [9.13 (d,  $J$  = 11.8 Hz), 8.81 (br. s), 1H], 8.53 (d,  $J$  = 11.8 Hz), 8.26 (d,  $J$  = 2.0 Hz), 1H], (7.47, 7.04, 6.85, 3m, 4H), (3.77, 3.74, 2s, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>), two rotamers.  $\delta$  163.6, 159.9, 157.5, 156.6, 130.3, 129.9, 122.1, 121.4, 116.9, 114.9, 114.2, 55.5, 55.4.

***N*-Cyclohexylformamide (3ei).**<sup>16</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 9:1) afforded the product as yellow solid. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.12 (s, 1H), 8.06 (s, 1H), 6.20 (broad s, 1H), 5.98 (broad s, 1H), 3.89-3.75 (m, 1H) and 3.35-3.22 (m, 1H), 1.94-1.08 (m, 10H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  163.8, 160.5, 51.2, 47.2, 34.8, 33.1, 25.5, 25.1, 24.9.

***N*-*p*-Tolylformamide (3ej).**<sup>28</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 95:5) afforded the product as yellow solid. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>), two rotamers (1:1)  $\delta$  8.97, 8.18 (2 br. s, 1H), [8.66 (d,  $J$  = 12.0 Hz), 8.33 (s), 1H], 7.48 (d,  $J$  = 8.5.0 Hz, 1H), 7.16-6.38 (m, 2H), 7.02 (d,  $J$  = 8.5.0 Hz, 1H), 2.37, 2.33 (2s, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>), two rotamers  $\delta$  163.3, 159.5, 135.0, 134.8, 130.3, 129.7, 120.4, 119.3, 20.9, 20.8.

***N*-Phenethylnicotinamide (3fh).**<sup>29</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 9:1) afforded the product as a white powder. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.88 (s, 1H), 8.58 (s, 1H), 8.05 (m, 1H), 7.71 (m, 7H), 3.69 (dd,  $J$  = 12.8, 6.9 Hz, 2H), 2.92 (t,  $J$  = 6.9 Hz, 2H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  166.0, 151.8, 147.9, 138.9, 135.4, 129.1, 126.6, 123.6, 41.6, 35.8.

***N*-Hexylnicotinamide (3fj).**<sup>30</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 9:1) afforded the product as a white powder. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.94 (m, 1H), 8.52 (br. s, 1H), 8.05 (m, 1H), 7.76 (m, 1H), 7.23 (m, 1H), 3.33 (dd,  $J$  = 13.3, 6.7 Hz, 2H), 1.51 (m, 2H), 1.19 (m, 6H), 0.76 (t,  $J$  = 6.6 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  165.8, 151.9, 148.3, 135.4, 130.9, 123.6, 40.3, 31.4, 29.4, 26.7, 22.5, 14.0.

***N*-Phenethylisonicotinamide (3gh).** Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 9:1) afforded the product as a white powder. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.67 (m, 2H), 7.53 (m, 2H), 7.29 (m, 5H), 6.63 (br. s, 1H), 3.75 (dd,  $J$  = 12.8, 6.9 Hz, 2H), 2.95 (t,  $J$  = 6.9 Hz, 2H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  165.5, 150.6, 138.5, 128.8, 121.3, 41.5, 35.3. HRMS (ESI+)  $m/z$  [M+H]<sup>+</sup> calcd for C<sub>14</sub>H<sub>15</sub>N<sub>2</sub>O 227.2817, found 227.2830.

***N*-Hexylisonicotinamide (3gj).**<sup>31</sup> Purification of the crude mixture by column chromatography (CH<sub>2</sub>Cl<sub>2</sub>:AcOEt 9:1) afforded the product as a white powder. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.61 (m, 2H), 7.62 (m, 2H), 7.29 (br. s, 1H), 3.38 (dd,  $J$  = 13.3, 6.7 Hz, 2H), 1.55 (m, 2H), 1.26 (m, 6H), 0.83 (t,  $J$  = 6.6 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  165.6, 150.4, 142.1, 121.4, 40.7, 31.8, 29.6, 27.0, 22.6, 14.1.

***N*<sup>1</sup>,*N*<sup>4</sup>-Dibenzylsuccinamide (3ha).**<sup>32</sup> Purification of the crude mixture by washing with CH<sub>2</sub>Cl<sub>2</sub> afforded the product as a white powder. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.35 (t,  $J$  = 5.2 Hz, 2H), 7.27 (m, 10H),

4.27 (d,  $J = 5.2$  Hz, 4H), 2.43 (s, 4H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  171.2, 139.5, 128.2, 127.1, 126.6, 40.0, 30.7.

***N*<sup>1</sup>,*N*<sup>4</sup>-Dicyclohexylsuccinamide (3hi).**<sup>33</sup> Purification of the crude mixture by washing with  $\text{CH}_2\text{Cl}_2$  afforded the product as gray powder.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.66 (m, 2H), 3.49 (m, 2H), 2.53 (4H), 1.72-1.40 (m, 10H), 1.31-0.99 (m, 10H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 47.7, 33.0, 31.7, 25.9, 25.0.

***N*<sup>1</sup>,*N*<sup>5</sup>-Dibenzylglutaramide (3ia).**<sup>32</sup> Purification of the crude mixture by washing with  $\text{CH}_2\text{Cl}_2$  afforded the product as a white powder.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.34 (br. s, 2H), 7.35-7.15 (m, 10H), 4.28 (d,  $J = 5.8$  Hz, 4H), 2.17 (t,  $J = 7.9$  Hz, 4H), 1.79 (m, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  171.2, 140.1, 128.8, 127.4, 127.2, 42.5, 35.3, 22.1.

**1,3-Dibenzylurea (3ja').**<sup>34</sup> Purification of the crude mixture by trituration ( $\text{H}_2\text{O}$  and heptane) afforded the product as white crystals.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35-7.20 (m, 10 H), 6.46 (t,  $J = 5.8$  Hz, 2H), 4.25 (d,  $J = 5.8$  Hz, 10 H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  158.5, 141.4, 128.7, 127.5, 127.0, 43.7.

**1,3-Bis(4-Methoxyphenyl)urea (3je).**<sup>35</sup> Purification of the crude mixture by trituration ( $\text{H}_2\text{O}$  and heptane) afforded the product as pale violet crystals.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.37 (broad s, 2H), 7.34 (d,  $J = 8.2$  Hz, 4H), 6.86 (d,  $J = 8.2$  Hz, 4H), 3.73 (s, 6H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  155.1, 153.7, 133.5, 120.6, 114.7, 55.9.

**1,3-Diphenethylurea (3jh).**<sup>36</sup> Purification of the crude mixture by trituration ( $\text{H}_2\text{O}$  and heptane) afforded the product as white crystals.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34-7.14 (m, 10H), 4.67 (t,  $J = 6.3$  Hz, 2H), 3.39 (dd,  $J = 13.3, 6.3$  Hz, 2H), 2.77 (t,  $J = 6.3$  Hz, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  158.2, 139.2, 128.8, 128.6, 41.7, 36.6.

**Tetrahydropyrimidin-2(1*H*)-one (3jk).**<sup>37</sup>  $^1\text{H}$  NMR (300 MHz,  $\text{DMSO}-d_6$ )  $\delta$  6.02 (s, 2H), 3.08 (m, 4H), 1.68 (m, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{DMSO}-d_6$ )  $\delta$  156.3, 39.8, 21.7.

**1-(2-Aminobenzyl)urea (3jl).** Purification of the crude mixture by trituration with water and heptane afforded the product as pale yellow solid.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.02-6.90 (m, 2H), 6.60 (m, 1H), 6.49 (m, 1H), 6.33 (t,  $J = 6.1$  Hz, 1H), 5.57 (broad s, 2H), 5.14 (broad s, 2H), 4.04 d,  $J = 6.1$  Hz, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  159.8, 147.1, 129.7, 128.5, 116.1, 115.4, 40.6. HRMS (ESI+)  $m/z$   $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_8\text{H}_{12}\text{N}_3\text{O}$  166.0980, found 166.0967.

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