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Hypervalent Iodine-Mediated Oxidative Rearrangement of N-H Ketimines: An Umpolung Approach to Amides

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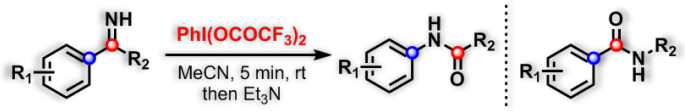
ABSTRACT: An umpolung approach to amides via hypervalent iodine-mediated oxidative rearrangement of N-H ketimines under mild reaction conditions is described. This strategy provides target amides with excellent selectivity in good yields. In addition, preliminary mechanistic studies demonstrated that the migration preference depends on both steric and electronic effect of the migrating groups.

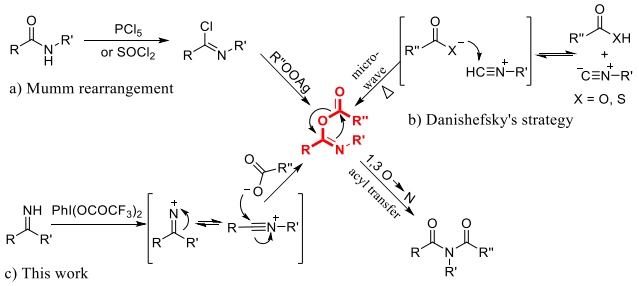


Amide bonds play important roles in life science and materials. It is reported that more than 25% pharmaceuticals on the market and 2/3 drug candidates contain amide bonds.[1] The conventional synthetic strategies for amide mainly relied on the activation of carboxylic acids and subsequent nucleophilic substitution by amines.[1a, 2] The development of highly efficient synthetic strategies for amide is always an important research topic for organic chemists from both industry and academia. After years of effort, the methods for preparing amide from chemicals other than carboxylic acids have been springing up.[3] Various carboxylic acid surrogates[4] have been demonstrated to be viable starting materials for amide bond formation. In addition, other strategies including Beckmann rearrangement,[5] Mumm rearrangement[6] and umpolung reactions[7] have been developed for amide synthesis. Among them, Mumm rearrangement, which involves an intramolecular 1,3 O→N acyl transfer of isoimide, is one of the oldest strategies to offer an amide bond (Scheme 1a).[6, 8] Recently, Danishefsky and coworkers developed a microwave-assisted coupling of carboxylic acid and isonitrile to furnish various *N*-formylamides via Mumm rearrangement (Scheme 1b).[9] The reaction proceeded smoothly at room temperature when thiocarboxylic acid was used in place of carboxylic acid.[9c] However, the inconvenient operation and nasty smell reagents were the concern for its further application. Mumm rearrangement with simple starting materials under mild reaction conditions still kept as a synthetic challenge. We herein described a hypervalent iodine reagent mediated umpolung approach to amide with Mumm rearrangement as the key step (Scheme 1c).

Umpolung reactions allow transformations, which otherwise do not work, to proceed by reversing the inherent polarity of one reactant.[10] It has been evolved into an efficient tool for organic chemists to realize difficult transformations in an uncommon manner.[10c, 10d] However, the application of umpolung reactions in amide bond construction is rare.[7b, 11] The inherent ability of λ^3-iodane reagent to act as an electrophile first and then as a leaving group enables it to serve as an umpolung reagent to convert an electron-rich center into an electron-deficient one.[12] It is noted that nitrilium species[13] is the key intermediate for Danishefsky's two-component coupling reaction to provide amide. We hypothesized that λ^3-iodanes mediated umpolung reactions of N-H ketimines would offer nitrilium, which could be used as valuable intermediate for further elaboration (Scheme 1c).

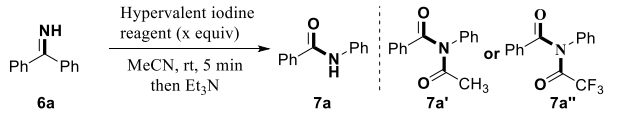
To test the validity of our hypothesis, we initiated from screening a series of λ^3-iodane oxidants with diphenyl methanimine (**6a**) as the model substrate. As shown in Table 1, no desired product was detected when λ^3-iodanes **1**, **2** and **3** was used

Scheme 1. Amide synthesis *via* 1, 3 O→N acyl migration



as oxidant (entries 1–3, Table 1). To our delight, moderate yield of mixture of amide **7a** and imidate **7a'** or **7a''** was obtained (entry 4, Table 1) when $PhI(OAc)_2$ **4** and diphenylmethanimine **6a** were stirred in acetonitrile for 5 min. Interestingly, **7a'** or **7a''** could transfer into **7a** with simple treatment of Et_3N. Further optimization disclosed that $PhI(OCOCF_3)_2$ **5** was the best choice of oxidant

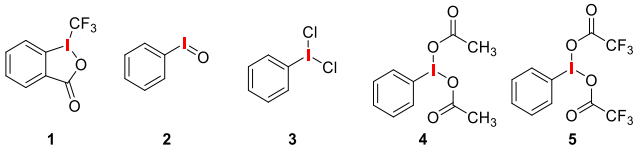
Table 1.Optimization of Reaction Conditions[a]



6a 7a 7a' 7a''

Entry	Hypervalent iodine reagent (x equiv)	Yield[b] (%)
1	**1** (2)	0
2	**2** (2)	0
3	**3** (2)	0
4	**4** (2)	65[c]
5	**5** (2)	96
6	**5** (1)	96
7	**5** (1)	96[d]
8	**5** (0.5)	40

[a]Reaction conditions: diphenylmethanimine **6a** (0.1 mmol), CH_3CN (2.0 mL), rt, 5 min, and Et_3N (4 equiv). [b]**7a'**:**7a** > 99:1 without Et_3N. [c]The reaction was performed under N_2 atmosphere.

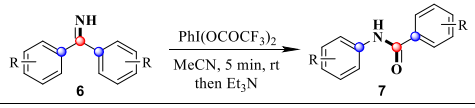


1 2 3 4 5

(entry 5, Table 1). Diphenylmethanimine **6a** could offer the amide **7a** in 96% with the consequent treatment of $PhI(OCOCF_3)_2$ (2 equiv) and Et_3N (4 equiv). Loading of $PhI(OCOCF_3)_2$ can be decreased to 1 equiv without affecting the reaction efficiency (entry 6, Table 1). Comparable excellent result was obtained when the reaction was performed under N_2 atmosphere and this result excluded the possibility that the oxygen atom of amide **7a** was originated from air (entry 7, Table 1).

To investigate the substrate scope of this methodology, we examined a series of symmetric diaryl N-H ketimines and the results are summarized in Table 2. A broad substrate scope was observed and all of the tested reaction proceeded smoothly to yield the target amide in good to excellent yields. Both the electron-donating groups (**7b–7f**) and electron-withdrawing groups (**7g–7i**) on the phenyl ring are tolerated. The methyl substituent at meta or para position showed no influence on the reaction efficiency (**7b, 7c**). Even the hindered ortho methyl group was compatible albeit slight lower yield was obtained (**7d**).

Table 2. Substrate scope of symmetric diaryl N-H ketimines[a]



6 7

7a, 97% **7b,** 96% **7c,** 96%

7d, 71% **7e,** 90% **7f,** 71%

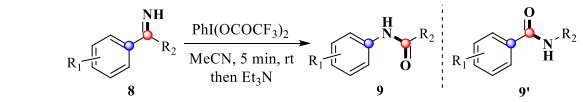
7g, 94% **7h,** 87% **7i,** 87%

[a]Reaction conditions: N-H ketimines (0.1 mmol), $PhI(OCOCF_3)_2$ (0.1 mmol), MeCN (2.0 mL), rt, 5 min, and Et_3N (4 equiv).

With the success on symmetric diaryl N-H ketimines in hand, we were encouraged to move toward more challenging unsymmetric ketimines, which would generate two isomers due to the two choices of migration (Table 3). To our delight, excellent aryl group migration selectivity was observed when alkylaryl N-H ketimines with linear alkyl substituent were treated with the standard reaction conditions (**9a-9g**). However, such selectivity was not always the case. A transition of migration preference from aryl functional group to alkyl one was observed when *α*-branched alkyl substituted alkylaryl N-H ketimines (**9i-9j**) were employed. Although cyclopropyl (**9h**) substituted phenyl N-H ketimine still offered the phenyl group migrated amide, alkyl group migrated amides were the major product for isobutyl (**9i'**) and cyclohexyl (**9j'**) ketimines. As an extreme, the alkyl group migrated amides are the only product for steric hindered alkyl ketimines such as adamantly (**9k**) and tert-butyl (**9l**) ketimines. Such migration preference transition might be attributed to both steric and electronic effect of the migrating group.

To further dissect the effects governing migration selectivity, diaryl N-H ketimines bearing an electron-withdrawing group on one aryl group and an electron-donating group on the other aryl moiety were employed as substrates (Scheme 2). The 4-MeO-, 4-F-, 4-Cl-, and 4-Br- substituted aryl ketimines were used as the

Table 3. Substrate scope of unsymmetric N-H ketimines[a]



8 9 9'

9a, 73% **9b,** 60% **9c,** 80 %

9d, 53% **9e,** 56% **9f,** 80%

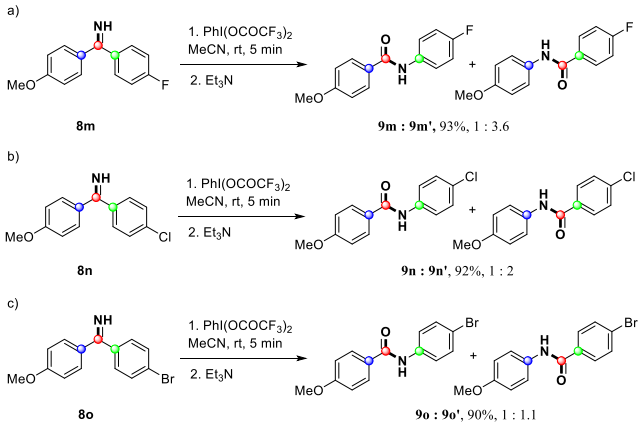
9g, 89% **9h,** 86% **9i : 9i',** 65%, 1:2[b]

9j : 9j', 64%, 1:2[b] **9k,** 53% **9l,** 65%

[a]Reaction conditions: N-H ketimines (0.1 mmol), $PhI(OCOCF_3)_2$ (0.1 mmol), MeCN (2 mL), rt, 5 min, and Et_3N (4 equiv). [b]The ratio was determined by NMR.

substituents due to their significant difference in electronegativity. In all cases,4-methoxylphenyl group migrated in preference to their halogen congeners, which unambiguously demonstrated that the migration was preferred to electron rich aryl group. Interestingly, the proportion of the products of 4-halophenyl migration increased gradually with a decrease in electronegativity of fluorine (**9m**), chlorine (**9n**) and bromine (**9o**) substituents. Thus, the ratio of the migrated products is consistent with the difference of electron density between the two migrating groups. The bigger the electron density difference the bigger the ratio of two migrated isomers.

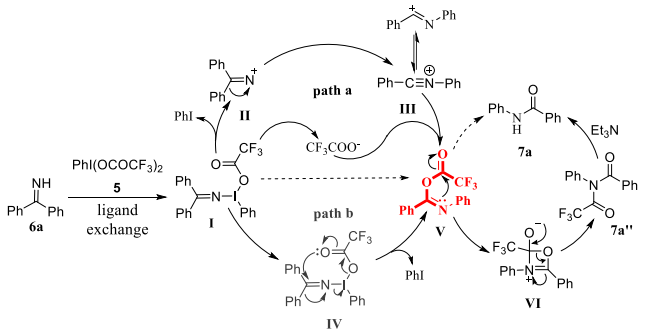
Scheme 2. The effects governing migration selectivity



It is noted that the *E*- and *Z*-isomers of acyclic ketimines could not be separated but transformed into each other even at low temperature.[14] In the present work, the *Z*- and *E*-isomers of 2, 2-dimethyl-1-phenylpropan-1-imine (**8l**) were obtained with a ratio of 3:1 (See SI for details). Only a single product **9l'** was obtained when the mixture of **8l** was treated with the standard reaction conditions. Thus, we believed that the migration selectivity is controlled by the electronic and steric effect of migrating group.

Two different pathways were proposed to rationalize the hypervalent iodine-mediated oxidative rearrangement of N-H ketimines (Scheme 3). The reaction was initiated by reacting **6a** with **5** to produce the intermediate **I** via ligand exchange, which may undergo reductive elimination of PhI to form the reactive species **II**. Followed by a 1, 2-shift to electron-deficient nitrogen atom, iminium **II** was converted to nitrilium **III**. **III** was then transformed into a "high-energy" intermediate **V** in the presence of trifluoroacetic acid anion[9a], which rearranged into the *N*-trifluoroacetyl amide **7a''** *via* 1,3 O→*N*-trifluoroacetyl transfer, which is similar with that of Mumm rearrangement (path a). Further treatment of **7a''** with Et_3N could selectively remove trifluoroacetyl group of **7a''** to offer **7a**. It is also possible that the intermediate **I** is converted into intermediate **V** through a concerted reductive elimination and an intramolecular rearrangements (path b). The intermediates **I**, **II**, **III** and **V** (or **7a''**) were detected by LC-MS. It should be noted that the ^{18}O labeled amide was detected when the reaction was carried out in ^{18}O-water (see SI for details). Although the stepwise path **a** is preferred, the concerted path **b** cannot be excluded at current stage.

Scheme 3. Possible Mechanism



In conclusion, we have developed the first hypervalent iodine-mediated umpolung approach to amide. The reaction proceeded efficiently under mild reaction conditions to provide target amides with excellent selectivity. The migration preference is related to the steric and electronic effect of the migrating groups. In addition, preliminary mechanism study demonstrated that a Mumm rearrangement would be involved in this reaction. This work offered a novel strategy to arylamides and paved the way for the application of hypervalent iodine reagent in amide synthesis.[15]

■ EXPERIMENTAL SECTION

All reactions were carried out in ovendried glassware. Anhydrous solvents were purified and dried by standard procedures. All commercially available reagents were used as received. All the reactions were monitored by thin-layer chromatography (TLC); products purification was done using silica gel column chromatography.

$^1H/^{13}C\{^1H\}$ NMR spectra were recorded on Bruker avance 400 MHz and Bruker AMX 400 MHz spectrometer at 400/100 MHz, respectively, in $CDCl_3$ and DMSO-d_6 unless otherwise stated, using either TMS or the undeuterated solvent residual signal as the reference. Chemical shifts are given in ppm and are measured relative to $CDCl_3$ and DMSO-d_6 as an internal standard. Mass spectra were obtained by the electrospray ionization time-of-flight (ESI-TOF) mass spectrometry. Flash column chromatography purification of compounds was carried out by gradient elution using ethyl acetate (EA) in light petroleum ether (PE). The N-H ketimines were prepared according to reported procedures.[16]

General procedure for the Preparation of N−H Ketimines 7a-k, 8h-k, and 8m-o[16]: In an ovendried Schlenk tube under N_2 atmosphere, a mixture of the aryl nitrile (10 mmol) and the aryl Grignard reagent (12 mmol) was stirred in THF (15 mL) at 80 °C for 12 h. The reaction mixture was cooled to room temperature and quenched by slow, dropwise addition of dry MeOH at 0 °C. After stirring vigorously for 0.5 h, the mixture was diluted with Et_2O (20 mL) and the resulting suspension was filtrated through a plug of triethylamine-deactivated silica gel. The volatile materials were evaporated under vacuum. The residue was further purified by flash column chromatography, with 1.5 mL of TEA added into every 100mL of eluent (PE/EA).

General procedure for the Preparation of N−H Ketimines 8a-g, and 8l[16]: A stirred solution of the aryl nitrile (10 mmol) in THF (15 mL) under a positive atmosphere of N_2 was cooled to -78 °C and the alkyllithium reagent (15 mmol) was added dropwise over 0.5 h. The mixture was stirred at -78 °C for 2 h, quenched with anhydrous MeOH (3 mL) and then stirred at room temperature for 2 h. The resulting suspension was filtrated through a plug of triethylamine-deactivated silica gel and the solvent was removed by rotary evaporation. The residue was purified by vacuum distillation.

(3r,5r,7r)-Adamantan-1-yl(phenyl)methanimine (8k) Purification by chromatography (petroleum ether/Et_3N = 100:1) afforded **8k** as a white solid (1.82 g, 76%); mp 53-55 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.05 (m, 5H), 2.07 – 1.95 (m, 5H), 1.85 (s, 4H), 1.68 (dd, *J* = 26.7, 11.8 Hz, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 190.1, 130.2, 128.0, 127.2, 126.6, 39.8, 39.2, 35.8, 28.2; HRMS-ESI (m/z): $[M+H]^+$ calcd for $C_{17}H_{21}N$: 240.1752; Found: 240.1750.

(4-Chlorophenyl)(4-methoxyphenyl)methanimine (8n) Purification by chromatography (petroleum ether/Et_3N = 100:1) afforded **8n** as a white solid (2.08 g, 85%); mp 95-96 °C; ^1H NMR (400 MHz, DMSO-d_6) δ 10.35 (brs, 1H), 7.55 – 7.46 (m, 6H), 6.99 (d, *J* = 8.7 Hz, 2H), 3.81 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, DMSO-d_6) δ 173.7, 160.8, 138.2, 134.5, 130.7, 129. 9, 129.7, 128.2, 113.7, 55.3; HRMS-ESI (m/z): $[M+H]^+$ calcd for $C_{14}H_{12}ClNO$: 246.0686; Found: 246.0684.

(4-Bromophenyl)(4-methoxyphenyl)methanimine (8o) Purification by chromatography (petroleum ether/Et_3N = 100:1) afforded **8o** as a white solid (2.49 g, 86%); mp 123-124 °C; ^1H NMR (400 MHz, DMSO-d_6) δ10.37 (brs, 1H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.47 (dd, *J* = 16.3, 8.5 Hz, 4H), 7.00 (d, *J* = 8.7 Hz, 2H), 3.81 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, DMSO-d_6) δ 173.8, 160.9, 138.5, 131.2, 130.6, 130.1, 129.7, 123.3, 113.7, 55.3; HRMS-ESI (m/z): $[M+H]^+$ calcd for $C_{14}H_{12}BrNO$: 290.0181; Found: 290.0179.

General procedure for amide synthesis: N−H Ketimines (0.1 mmol) was slowly added to a solution of iodobenzenedi(trifluoroacetate) (0.1 mmol, 43.0 mg) in anhydrous acetonitrile (2 mL) at room temperature under magnetic stirring. The reaction mixture was stirred at room temperature for 5 min. After the reaction was completion (TLC), trimethylamine (0.5 mmol) or sodium methoxide (0.5 mmol in 0.5 mL methanol) were added. The reaction mixture was stirred at room temperature for 0.5~3 h. The solvent was then removed in vacuo and the product was isolated by column chromatography on silica gel.

N-phenylbenzamide (7a)[17] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **7a** as a white solid (19.1 mg, 97%); mp 163-164 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.97 (s, 1H), 7.86 (d, *J* = 7.2 Hz, 2H), 7.65 (d, *J* = 7.7 Hz, 2H), 7.55 – 7.52 (m, 1H), 7.46 (t, *J* = 7.4 Hz, 2H), 7.36 (t, *J* = 7.7 Hz, 2H), 7.17 – 7.13 (m, 1H);$^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 166.0, 138.1, 135.1, 131.9, 129.2, 128.9, 127.2, 124.7, 120.4.

N-acetyl-N-phenylbenzamide (7a')[18] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **7a'** as a white solid (15.5 mg, 65%); mp 70-71 °C; ^1H NMR (400 MHz, $CDCl_3$)

δ 7.53 (d, *J* = 7.3 Hz, 2H), 7.35 – 7.31 (m, 1H), 7.28 – 7.18 (m, 5H), 7.08 (d, *J* = 7.4 Hz, 2H), 2.35 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 173.6, 173.0, 139.3, 135.0, 132.2, 129.5, 129.4, 128.7, 128.4, 128.2, 25.8.

4-Methyl-N-(p-tolyl)benzamide (7b)[19] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **7b** as a white solid (20.6 mg, 96%); mp 210-211 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.67 (d, *J* = 7.4 Hz, 2H), 7.44 (d, *J* = 7.6 Hz, 2H), 7.17 (d, *J* = 7.5 Hz, 2H), 7.07 (d, *J* = 7.5 Hz, 2H), 2.33 (s, 3H), 2.25 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 165.8, 142.3, 135.6, 134.2, 132.3, 129.6, 129.5, 127.1, 120.4, 21.6, 21.0.

3-Methyl-N-(m-tolyl)benzamide (7c)[20] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **7c** as a white solid (21.6 mg, 96%); mp 125-126 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (brs, 1H), 7.66 – 7.63 (m, 2H), 7.50 (s, 1H), 7.43 – 7.41 (m, 1H), 7.33 (s, 1H), 7.25 – 7.21 (m, 1H), 6.96 – 6.94 (m, 1H), 2.40 (s, 3H), 2.35 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 166.1, 139.1, 138.7, 138.1, 135.2, 132.6, 129.0, 128.7, 127.9, 125.4, 124.1, 121.0, 117.4, 21.6, 21.5.

2-Methyl-N-(o-tolyl)benzamide (7d) [21] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **7d** as a white solid (16 mg, 71%); mp 141-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.90 (m, 1H), 7.45 – 7.44 (m, 1H), 7.32 – 7.28 (m, 1H), 7.22 – 7.15 (m, 5H), 7.05 (t, *J* = 7.2 Hz, 1H), 2.46 (s, 3H), 2.23 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 168.2, 136.7, 135.9, 131.5, 130.7 (2C), 130.4, 129.2, 127.0, 126.8, 126.1, 125.5, 123.2, 20.1, 18.1.

3-Methoxy-N-(3-methoxyphenyl)benzamide (7e)[22] Purification by chromatography (petroleum ether/ EtOAc = 4:1) afforded **7e** as a white solid (23.1 mg, 90%); mp 96-97 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.89 (m, 1H), 7.43 – 7.42 (m, 2H), 7.37 – 7.35 (m, 2H), 7.26 – 7.22 (m, 1H), 7.10 – 7.06 (m, 2H), 6.70 (dd, *J* = 8.2, 2.2 Hz, 1H), 3.85-3.84 (m, 3H), 3.82-3.81 (m, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 165.7, 160.4, 160.2, 139.3, 136.6, 129.9 (2C), 118.8, 118.2, 112.6, 112.4, 110.7, 106.0, 55.6, 55.5.

4-Methoxy-N-(4-methoxyphenyl)benzamide (7f)[23] Purification by chromatography (petroleum ether/ EtOAc = 2:1) afforded **7f** as a white solid (18.2 mg, 71%); mp 206-207 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.97 (s, 1H), 7.94 (d, *J* = 8.5 Hz, 2H), 7.65 (d, *J* = 8.7 Hz, 2H), 7.05 (d, *J* = 8.5 Hz, 2H), 6.91 (d, *J* = 8.7 Hz, 2H), 3.83 (s, 3H), 3.74 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ 164.5, 161.8, 155.4, 132.4, 129.5, 127.1, 122.0, 113.7, 113.6, 55.4, 55.2.

4-Fluoro-N-(4-fluorophenyl)benzamide (7g)[19] *Purification* by chromatography (petroleum ether/ EtOAc = 6:1) afforded **7g** as a white solid (21.9 mg, 94%); mp 180-181 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.32 (s, 1H), 8.05 – 8.01 (m, 2H), 7.79 – 7.76 (m, 2H), 7.39 – 7.34 (m, 2H), 7.21 – 7.17 (m, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ 165.3, 163.6 (d, *J* = 151.1 Hz), 158.4 (d, *J* = 240.4 Hz), 135.4, 131.2 (d, *J* = 3.0 Hz), 130.4 (d, *J* = 9.1 Hz), 122.3 (d, *J* = 7.8 Hz), 115.3 (dd, *J* = 22.0, 15.3 Hz, 2C).

4-Chloro-N-(4-chlorophenyl)benzamide (7h)[19] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **7h** as a white solid (23.1 mg, 87%); mp 202-203 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.44 (s, 1H), 7.98 (d, *J* = 8.4 Hz, 2H), 7.81 (d, *J* = 8.7 Hz, 2H), 7.62 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.7 Hz, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ 164.5, 138.0, 136.6, 133.4, 129.7, 128.6, 128.5, 127.5, 121.9.

4-Bromo-N-(4-bromophenyl)benzamide (7i)[23] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **7i** as a white solid (30.6 mg, 87%); mp 223-224 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.44 (s, 1H), 7.90 (d, *J* = 8.5 Hz, 2H), 7.75 – 7.74 (m, 4H), 7.53 (d, *J* = 8.7 Hz, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 164.7, 138.4, 133.7, 131.5 (2C), 129.8, 125.5, 122.3, 115.6.

N-phenylpentanamide (9a) [4d] Purification by chromatography (petroleum ether/ EtOAc = 5:1) afforded **9a** as a white solid (12.9 mg, 73%); mp 59-60 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.9 Hz, 1H), 7.30 (t, *J* = 7.7 Hz, 1H), 7.09 (t, *J* = 7.3 Hz, 1H), 2.35 (t, *J* = 7.6 Hz, 1H), 1.77 – 1.65 (m, 1H), 1.46 – 1.32 (m, 1H), 0.93 (t, *J* = 7.3 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 171.8, 138.2, 129.0, 124.3, 120.1, 37.6, 27.8, 22.5, 13.9.

N-(p-tolyl)pentanamide (9b)[19] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9b** as a white solid (11.5 mg, 60%); mp 67-68 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.38 (m, 2H), 7.19 (brs, 1H), 7.12 – 7.10 (m, 2H), 2.36 – 2.30 (m, 5H), 1.72 – 1.68 (m, 2H), 1.41 – 1.38 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 171.5, 135.5, 133.9, 129.6, 120.0, 37.7, 27.9, 22.5, 21.0, 14.0.

N-(4-methoxyphenyl)pentanamide (9c)[32] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9c** as a white solid (16.6 mg, 80%); mp 79-80 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (s, 1H), 7.41 (d, *J* = 8.9 Hz, 2H), 6.82 (d, *J* = 8.9 Hz, 2H), 3.77 (s, 3H), 2.31 (t, *J* = 7.6 Hz, 2H), 1.68 – 1.66 (m, 2H), 1.38 – 1.35 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 171.6, 156.3, 131.2, 121.9, 114.0, 55.6, 37.4, 27.9, 22.5, 14.0.

N-(4-fluorophenyl)pentanamide (9d)[17b] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9d** as a white solid (10.3 mg, 53%); mp 50-51 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.44 (m, 2H), 7.00 – 6.96 (m, 2H), 2.33 (t, *J* = 7.5 Hz, 1H), 1.77 – 1.63 (m, 1H), 1.41– 1.36 (m, 2H), 0.93 (t, *J* = 7.2 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 171.7, δ 159.5 (d, J = 243.4 Hz), 134.1, 121.9 (d, J = 7.6 Hz), 115.7 (d, J = 21.9 Hz), 37.5, 27.8, 22.5, 13.9.

N-([1,1'-biphenyl]-4-yl)pentanamide (9e)[30] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9e** as a white solid (14.2 mg, 56%); mp 169-170 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 – 7.53 (m, 6H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.32 (t, *J* = 7.3 Hz, 2H), 2.38 (t, *J* = 7.6 Hz, 2H), 1.77 – 1.70 (m, 2H), 1.47 – 1.38 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 171.7, 140.7, 137.5, 137.2, 128.9, 127.7, 127.0, 120.3, 37.7, 27.9, 22.5, 13.9.

N-(naphthalen-2-yl)pentanamide (9f)[31] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9f** as a white solid (18.2 mg, 80%); mp 84-85 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (s, 1H), 7.79 – 7.77 (m, 3H), 7.47 – 7.38 (m, 3H), 7.30 (brs, 1H), 2.42 (t, *J* = 7.5 Hz, 2H), 1.78 – 1.74 (m, 2H), 1.47 – 1.44 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 171.5, 135.4, 133.9, 130.6, 128.7, 127.6, 127.5, 126.5, 124.9, 119.8, 116.5, 37.6, 27.7, 22.4, 13.8.

3-Methyl-N-phenylbutanamide (9g)[26] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9g** as a white solid (15.8 mg, 89%); mp 112-113 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.50 (m, 2H), 7.33 – 7.29 (m, 2H), 7.21 (brs, 1H), 7.10 (t, *J* = 7.3 Hz, 1H), 2.22 (s, 3H), 1.02 (d, *J* = 5.8 Hz, 6H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 170.9, 138.1, 129.1, 124.4, 120.0, 47.3, 26.4, 22.6.

N-phenylcyclopropanecarboxamide (9h)[22] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9h** as a white solid (13.8 mg, 86%); mp 111-112 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.51 (d, *J* = 7.8 Hz, 2H), 7.30 – 7.26 (m, 2H), 7.07 (t, *J* = 7.2 Hz, 1H), 1.25 (s, 1H), 1.06 – 1.05 (m, 2H), 0.82 – 0.80 (m, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 172.3, 138.3, 129.0, 124.1, 119.9, 15.8, 8.1.

2-Methyl-N-phenylbutanamide (9i)[25] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9i** as a white solid (3.8 mg, 22%); mp 95-96 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54– 7.52 (m, 2H), 7.33 – 7.29 (m, 2H), 7.17 (brs, 1H), 7.09 (t, *J* = 7.3 Hz, 1H), 2.29 – 2.22 (m, 1H), 1.83 – 1.72 (m, 1H), 1.56 – 1.47 (m, 1H), 1.23 (d, *J* = 6.8 Hz, 3H), 0.97 (t, *J* = 7.4 Hz, 3H);

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 174.7, 138.0, 129.0, 124.2, 119.8, 44.3, 27.4, 17.5, 11.9.

N-(sec-butyl)benzamide (9i')[24] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9i'** as a white solid (7.7 mg, 43%); mp 89-90 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.6 Hz, 2H), 7.49 – 7.45 (m, 1H), 7.42 – 7.38 (m, 2H), 5.99 (brs, 1H), 4.15 – 4.08 (m, 1H), 1.61 – 1.53 (m, 2H), 1.22 (d, *J* = 6.6 Hz, 3H), 0.96 (t, *J* = 7.4 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 167.0, 135.3, 131.3, 128.6, 126.9, 47.2, 29.9, 20.6, 10.5.

N-phenylcyclohexanecarboxamide (9j)[17a] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9j** as a white solid (4.3 mg, 21%); mp 147-148 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.50 (m, 2H), 7.32 – 7.28 (m, 2H), 7.14 (brs, 1H), 7.09 (t, *J* = 7.3 Hz, 1H), 2.25 – 2.20 (m, 1H), 1.98 – 1.94 (m, 2H), 1.85 – 1.83 (m, 2H), 1.72 – 1.70 (m, 1H), 1.53 – 1.50 (m, 1H), 1.36 – 1.24 (m, 4H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 174.2, 138.1, 129.0, 124.1, 119.8, 46.6, 29.7, 25.7.

N-cyclohexylbenzamide (9j') [28] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9j'** as a white solid (8.7 mg, 43%); mp 144-146 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 – 7.73 (m, 2H), 7.48 (t, *J* = 7.3 Hz, 1H), 7.44 – 7.40 (m, 2H), 6.01 – 5.96 (m, 1H), 4.02 – 3.93 (m, 1H), 2.04 – 2.01 (m, 2H), 1.78 – 1.73 (m, 2H), 1.49 – 1.39 (m, 2H), 1.28 – 1.19 (m, 4H); $^{13}C\{^{1}H\}$ NMR (100MHz, $CDCl_3$) δ 166.6, 135.1, 131.3, 128.5, 126.8, 48.7, 33.3, 25.6, 24.9.

N-((1R,3s)-adamantan-1-yl)benzamide (9k)[29] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9k** as a white solid (13.5 mg, 53%); mp 159-160 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.70 (m, 2H), 7.48 – 7.38 (m, 3H), 5.81 (brs, 1H), 2.12 (s, 9H), 1.72 (s, 6H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 166.2, 135.5, 130.6, 128.0, 126.2, 51.8, 41.2, 35.9, 29.0.

N-(tert-butyl)benzamide (9l)[27] Purification by chromatography (petroleum ether/ EtOAc = 6:1) afforded **9l** as a white solid (11.5 mg, 65%); mp 133-134 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.1 Hz, 2H), 7.48 – 7.45 (m, 1H), 7.42-7.38 (m, 2H), 1.47 (s, 9H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 167.1, 136.2, 131.2, 128.6, 126.8, 51.7, 29.0.

N-(4-fluorophenyl)-4-methoxybenzamide (9m)[33] Purification by chromatography (petroleum ether/ EtOAc = 8:1) afforded **9m** as a white solid (5 mg, 36%); mp 190-191 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.11 (s, 1H), 7.95 (d, *J* = 8.9 Hz, 2H), 7.79 – 7.75 (m, 2H), 7.19 – 7.15 (m, 2H), 7.07 – 7.05(m, 2H), 3.84 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ 164.8, 161.9, 129.5 (2C), 122.1 (d, *J* = 7.7 Hz), 115.1 (d, *J* = 22.1 Hz), 113.6 (2C), 55.4.

4-Fluoro-N-(4-methoxyphenyl)benzamide (9m')[33] Purification by chromatography (petroleum ether/ EtOAc = 8:1) afforded **9m'** as a white solid (17.8 mg, 57%); mp 196-197 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.12 (s, 1H), 8.05 – 8.01 (m, 2H), 7.66 (d, *J* = 8.8 Hz, 2H), 7.34 (t, *J* = 8.7 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 3.75 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ 164.6 (d, *J* = 121.8 Hz), 162.7, 155.6, 132.1, 131.4 (d, *J* = 2.9 Hz), 130.2 (d, *J* = 9.0 Hz), 122.0, 115.2 (d, *J* = 21.8 Hz), 113.7, 55.1.

N-(4-chlorophenyl)-4-methoxybenzamide (9n)[34] Purification by chromatography (petroleum ether/ EtOAc = 10:1) afforded 9n as a white solid (18.5 mg, 32%); mp 208-209 °C; 1H NMR (400 MHz, CDCl3) δ 10.31 (s, 1H), 7.98 (d, J = 8.8 Hz, 2H), 7.83 (d, J = 8.9 Hz, 2H), 7.38 (d, J = 8.8 Hz, 2H), 7.05 (d, J = 8.8 Hz, 2H), 3.83 (s, 3H); 13C{^{1}H} NMR (100 MHz, DMSO-d6) δ 170.2, 167.2, 143.8, 134.9, 133.6, 132.2, 131.9, 127.1, 118.8, 60.7.

4-Chloro-N-(4-methoxyphenyl)benzamide (9n')[34] Purification by chromatography (petroleum ether/ EtOAc = 10:1) afforded **9n'** as a white solid (16 mg, 60%); mp 208-209 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.20 (s, 1H), 7.97 (d, *J* = 8.6 Hz, 2H), 7.65 (d, *J* = 9.0 Hz, 2H), 7.60 (d, *J* = 8.6 Hz, 2H), 6.93 (d, *J* = 9.1 Hz, 2H), 3.74 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ 164.0, 155.7, 136.2, 133.7, 132.0, 129.5, 128.4, 122.0, 113.8, 55.2.

N-(4-bromophenyl)-4-methoxybenzamide (9o)[35] Purification by chromatography (petroleum ether/ EtOAc = 10:1) afforded 9o as a white solid (13.2 mg, 43%); mp 206-208 °C; 1H NMR (400 MHz, DMSO-d6) δ 10.30 (s, 1H), 7.97 (d, J = 8.8 Hz, 2H), 7.77 (d, J = 8.8 Hz, 2H), 7.51 (d, J = 8.8 Hz, 2H), 7.05 (d, J = 8.8 Hz, 2H), 3.83 (s, 3H); 13C{^{1}H} NMR (100 MHz, DMSO-d6) δ 165.0, 162.0, 138.8, 131.3, 129.7, 126.7, 122.2, 115.0, 113.6, 55.4.

4-Bromo-N-(4-methoxyphenyl)benzamide (9o')[36] Purification by chromatography (petroleum ether/ EtOAc = 10:1) afforded **9o'** as a white solid (14.4 mg, 47%); mp 222-223 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.17 (s, 1H), 7.90 (d, *J* = 8.3 Hz, 2H), 7.73 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 3.74 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ 164.0, 155.6, 134.1, 131.9, 131.3, 129.6, 125.0, 122.0, 113.7, 55.2.

■ ASSOCIATED CONTENT

Supporting Information

The copies of LC-MS (ESI) of control experiments and the copies of ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra are available free of charge via the internet at http://pubs.acs.org.

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Notes

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