Copper-Catalyzed Cross-Coupling of *O*-Alkyl Hydroxamates with Aryl Iodides

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Abstract: *N*-Aryl-*O*-alkylhydroxamic acid derivatives were prepared by copper-catalyzed cross-coupling of hydroxamates with aryl iodides. The reaction conditions are compatible with standard hydroxy-protecting groups on the hydroxylamine moiety and are applicable to a broad range of coupling partners.

Key words: copper, arylation, cross-coupling, amides, halides, catalysis

N-Arylhydroxamic acids are widely used in many areas of organic[1–12] and medicinal chemistry.[13–17] For example, their derivatives are intermediates in the preparation of nitrogen heterocycles,[1–6] 2-aminophenols,[7] thiohydroxamic acids,[8] and chiral *N*-arylaziridines,[9] as well as being substrates for enantioselective Diels−Alder reactions.[10] *N*-Arylhydroxamic acids are N−OH mediators for biocatalytical processes,[11] and their complexes with tungsten, molybdenum, or vanadium are used as catalysts in various oxidation reactions.[12] In addition, *N*-arylhydroxamic acids serve as *N*,*O*-acyltransferase substrates,[13] possess antiinflammatory,[14] antiviral, cytostatic,[15] and antioxidant properties,[16] and they have been tested as glyoxalase inhibitors.[17]

N-Arylhydroxamic acids are usually synthesized by acylation of arylhydroxylamines,[9b,18] by oxidation of arylacylamides,[19] or by the reaction of aryl nitroso compounds with aldehydes.[5c,15,20] Two methods have recently been developed for the synthesis of cyclic *N*-arylhydroxamic acids by palladium-catalyzed C–H bond activation.[21]

Surprisingly, transition metal-catalyzed C–N cross-coupling, a reaction that is routinely employed for N-arylation of amides,[22] sulfonamides,[23] sulfoximines,[24] sulfonimidamides,[25] hydrazides,[26] hydrazones,[27] or hydrazines,[28] has not been applied to the intermolecular arylation of hydroxamic acid derivatives. Only O-substituted hydroxylamine carbamates have been shown to be suitable substrates for this transformation.[29]

We recently reported a new approach for the synthesis of *N*-alkoxyindol-2-ones.[30] The key step for this process is the intramolecular N-arylation of 2-(2-bromoaryl)acetylhydroxamates in the presence of copper(II) bromide and 20 mol% of *N*,*N*′-dimethylethylenediamine (DMEDA). We surmised that the catalytic system that we developed might also be suitable for intermolecular N-arylation of hydroxamic acids, and we used the conditions for the intramolecular reaction as a starting point for the development of an intermolecular version of this transformation.

Our initial attempts to react hydroxamate **1a** with iodobenzene at 80 °C for 18 hours gave the *N*-phenylhydroxamic acid derivative **2a** in 60% yield (Table 1, entry 1). After examining numerous combinations of the copper source, ligand, base, and solvent (see Supporting Information), we found that replacing copper(II) bromide with copper(I) oxide was the only modification of the initial reaction conditions that enhanced the yield of the coupling product **2a**.

We then applied this improved reaction protocol to the cross-coupling of various hydroxamic acid derivatives with iodobenzene. Standard hydroxy-protecting groups on the hydroxylamine moiety were well tolerated (Table 1, entries 2–5). However, it was necessary to increase the reaction temperature to 110 °C to achieve full conversion of the bulkier *tert*-butyl hydroxamate **1e**. Protection of the hydroxy group on the hydroxylamine moiety was essential. In the case of the hydroxamic acid **1f**, only phenylacetamide and a trace amount of the N-arylated product were detected in the product mixture (entry 6). Phenylacetylhydroxamates **1g** and **1h**, which are substituted at the α-position were also suitable substrates for the N-arylation reaction (entries 7 and 8). *N*-Phenyl derivatives **2i**–**k** were obtained in lower yields; however, the outcome was improved by increasing the reaction times (entries 10 and 11). The yields of aryl and hetaryl *N*-phenylhydroxamates **2l**–**r** were also moderate (entries 12–18). However, it should be pointed out that a previous attempt to cross-couple *N*-alkoxybenzamides with aryl halides was unsuccessful.[29b]

Having evaluated the range of hydroxamates that are suitable coupling partners, we examined the efficiency of cross-coupling of various aryl halide components (Table 2). The method was efficient for a wide range of aryl iodides, affording the corresponding products in high-to-excellent yields (82–97%). The arylation conditions are compatible with various functional groups in the substrate, including ester, nitro, keto, and nitrile groups (entries 1–8, 20). The presence of an electron–donating group in the *para*-position with respect to iodine only slightly reduced the yield (71–79%) of the corresponding *N*-arylhydroxamate (entries 11–14). The developed reaction conditions are, however, unsuitable for N-arylation with

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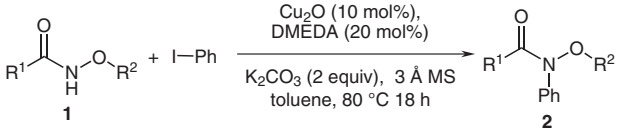
aryl bromides or chlorides. Even at elevated temperatures and with prolonged reaction times, the starting hydroxamate **1a** remained almost intact. As a consequence, bromo- or chloro-substituted iodobenzenes can be used as coupling partners to give the corresponding halogenated *N*-arylhydroxamates **3l**–**n** in good yields (entries 18–20). Furthermore, the method is not restricted to iodoarenes, as iodopyridines also react to form the corresponding N-substituted pyridyl derivatives (entries 21 and 22).

Table 1 N-Arylation of Various Hydroxamates with Iodobenzene



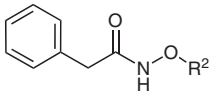
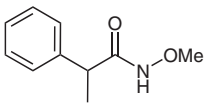
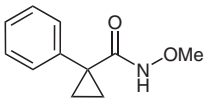
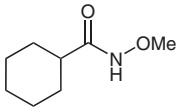
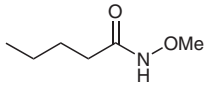
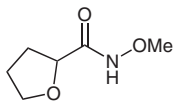
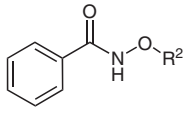
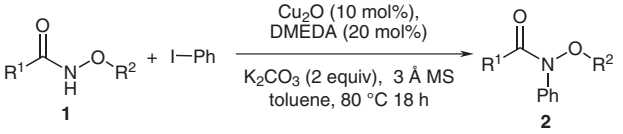
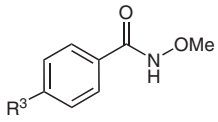
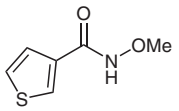
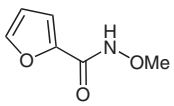
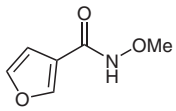
Entry[a]		Reactant	Product	Yield (%)
1	**1a**		**2a**	88 (60[b], 90[c])
		R^2 = Me		
2	**1b**	R^2 = allyl	**2b**	96
3	**1c**	R^2 = Bn	**2c**	71
4	**1d**	R^2 = THP	**2d**	81
5	**1e**	R^2 = *t*-Bu	**2e**	73[d]
6	**1f**	R^2 = H	**2f**	0
7	**1g**		**2g**	82
8	**1h**		**2h**	70[d]
9	**1i**		**2i**	63 (66[c])
10	**1j**		**2j**	61 (95[e])
11	**1k**		**2k**	27 (41[e])
12	**1l**		**2l**	35
		R^2 = Me		
13	**1m**	R^2 = allyl	**2m**	44

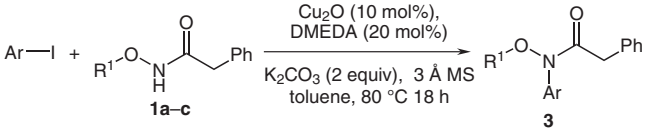
Table 1 N-Arylation of Various Hydroxamates with Iodobenzene (continued)



Entry[a]		Reactant	Product	Yield (%)
14	**1n**		**2n**	26 (20[c])
		R^3 = OMe		
15	**1o**	R^3 = CF_3	**2o**	60
16	**1p**		**2p**	43 (21[b], 64[e])
17	**1q**		**2q**	51
18	**1r**		**2r**	72

[a] Reaction conditions: hydroxamate **1** (0.2 mmol), PhI (1.5 equiv), Cu_2O (10 mol%), DMEDA (20 mol%), K_2CO_3 (2.0 equiv), 3 Å MS (100% of weight), toluene, 80 °C, 18 h.
[b] $CuBr_2$ instead of Cu_2O.
[c] 1.0 mmol scale.
[d] At 110 °C.
[e] After 36 h.

Table 2 N-Arylation of 2-Phenylacetylhydroxamates with Various Aryl Iodides



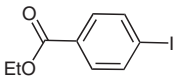
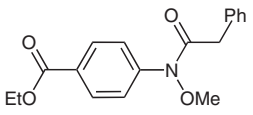
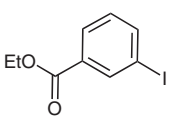
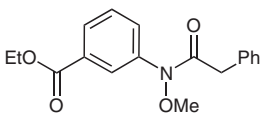
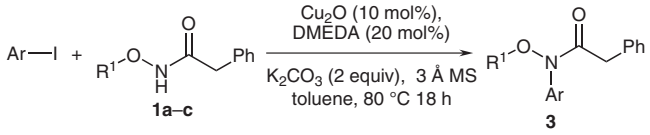
Entry[a]	Aryl iodide	Product **3**	Yield (%)
1		 **3a**	90
2		 **3b**	92

Table 2 N-Arylation of 2-Phenylacetylhydroxamates with Various Aryl Iodides (continued)



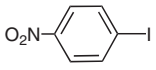
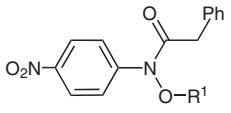
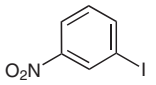
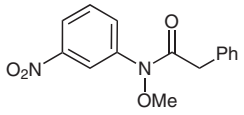
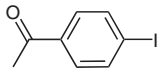
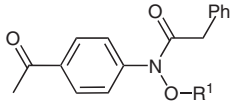
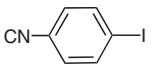
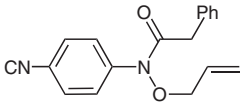
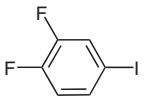
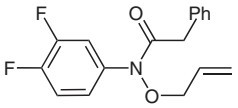
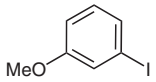
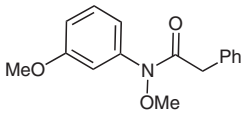
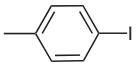
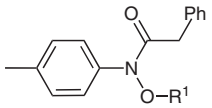
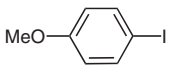
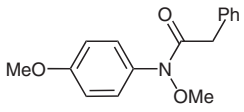
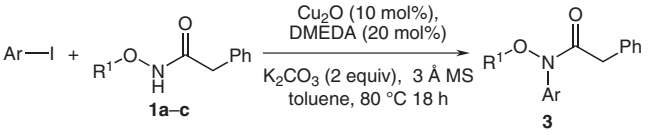
Entry[a]	Aryl iodide	Product **3**	Yield (%)
3		 **3c1** R^1 = Me	92
4		**3c2** R^1 = allyl	90
5		 **3d**	82
6		 **3e1** R^1 = Me	89
7		**3e2** R^1 = allyl	84 (90[b])
8		 **3f**	90
9		 **3g**	93
10		 **3h**	97
11		 **3j1** R^1 = Me	71
12		**3j2** R^1 = allyl	79
13		**3j3** R^1 = Bn	79
14		 **3k**	72 (87[b])
15	PhCl	**2a**	0

Table 2 N-Arylation of 2-Phenylacetylhydroxamates with Various Aryl Iodides (continued)



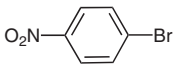
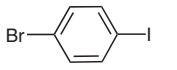
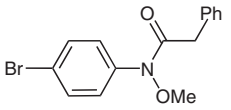
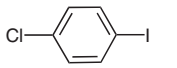
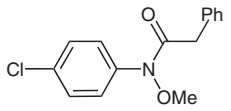
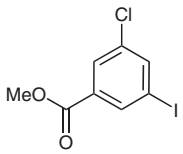
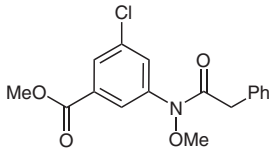
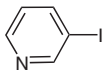
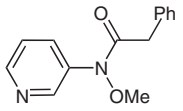
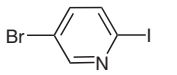
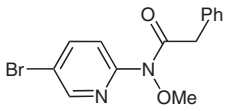
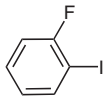
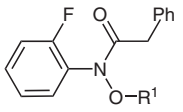
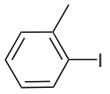
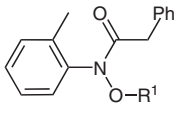
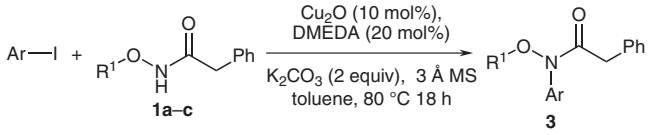
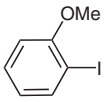
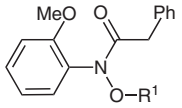
Entry[a]	Aryl iodide	Product **3**	Yield (%)
16	PhBr	**2a**	0
17		**3c1**	0
18		 **3l**	75
19		 **3m**	82
20		 **3n**	88
21		 **3o**	89 (88[b])
22		 **3p**	63
23		 **3q1** R^1 = Me	38
24		**3q2** R^1 = allyl	63
25		**3q3** R^1 = Bn	48 (40[b])
26		 **3r1** R^1 = Me	24
27		**3r2** R^1 = allyl	35

Table 2 N-Arylation of 2-Phenylacetylhydroxamates with Various Aryl Iodides (continued)



Entry[a]	Aryl iodide	Product **3**	Yield (%)
28		 **3s1** R^1 = Me	0
29		**3s2** R^1 = allyl	38
30		**3s3** R^1 = Bn	24

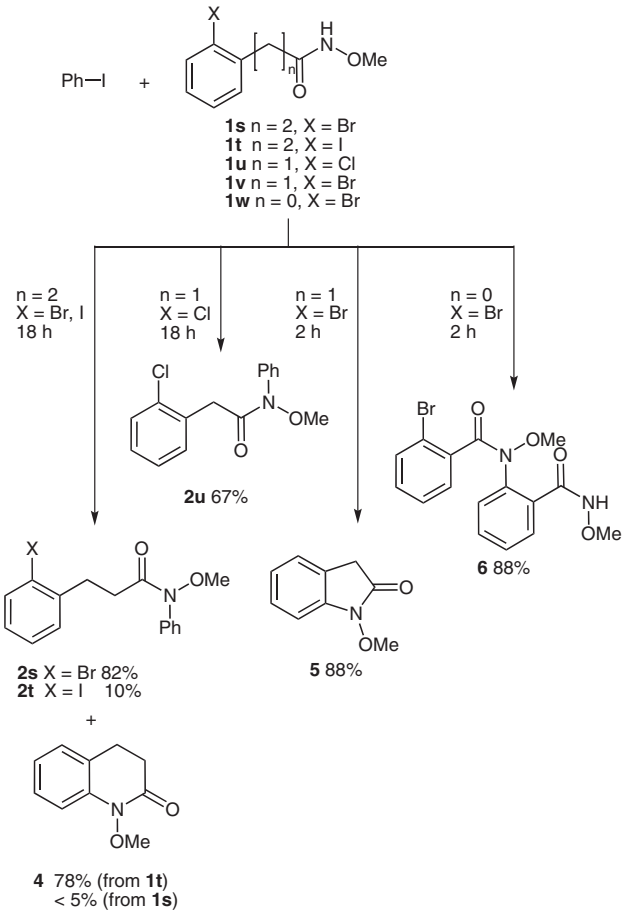
[a] Reaction conditions: hydroxamate **1a–c** (0.2 mmol), (het)aryl iodide (1.5 equiv), Cu_2O (10 mol%), DMEDA (20 mol%), K_2CO_3 (2.0 equiv), 3 Å MS (100% of weight), toluene, 80 °C, 18 h.
[b] 1.0 mmol scale.

Another class of challenging substrates are the *ortho*-substituted iodoarenes, because it has been reported[29a] that copper-catalyzed cross-coupling of hydroxylamine carbamates with 2-iodotoluene does not afford the corresponding N-arylated product.[31] Indeed, the *N*-arylhydroxamates **3q**–**s** gave gave low-to-moderate yields of the corresponding products under our reaction conditions (entries 23–30), and the 2-methoxy derivative (entry 28) was not obtained at all.

Finally, we subjected several derivatives of hydroxamic acid bearing an aryl halogen moiety (which are formally suitable for intramolecular arylation) to the current reaction condition in the presence of iodobenzene (1.5 equivalents) (Scheme 1). As expected, the indolone **5** was the sole product in the reaction of hydroxamate **1v**. An intramolecular reaction with the formation of quinolinone **4** also predominated in the case of hydroxamate **1t**. However, the related chloro- and bromo hydroxamates **1u** and **1s**, respectively, gave the corresponding *N*-phenyl derivatives **2s** and **2u** in >65% yield.

More surprising was the outcome of the reaction with hydroxamate **1w**. In this case, the homocoupled product **6** (Figure 1) was isolated exclusively in 88% yield after two hours at 80 °C.

Aryl bromides, including 1-bromo-4-nitrobenzene (Table 2, entry 17), were inefficient coupling partners, and benzohydroxamates were arylated in moderate yields, whereas the cyclization of bromo hydroxamate **1v** to give the indolone **5** proceeded very efficiently in two hours. We therefore assume that formation of **6** might proceed in a pseudo-intramolecular fashion. The most widely accepted mechanism[32] for the copper-catalyzed N-arylation of amides involves the copper amidate intermediate **7** (Scheme 2). However, the presence of an inactive cuprate complex **8** in the reaction medium has also been postulated.[32a,b] In



Scheme 1 *Reagents and conditions*: PhI (1.5 equiv), Cu_2O (10 mol%), DMEDA (20 mol%), K_2CO_3 (2.0 equiv), 3 Å MS (100% of weight), toluene, 80 °C.

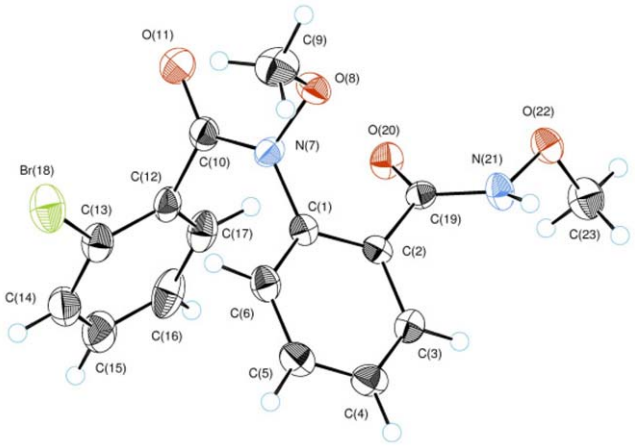
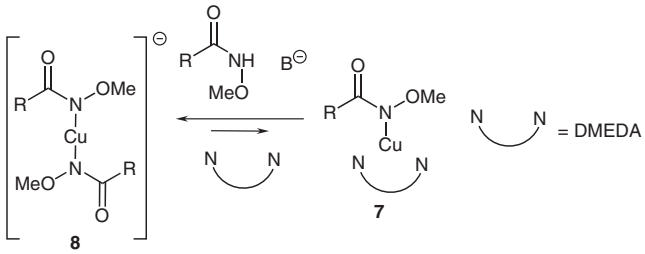


Figure 1 X-ray crystal structure of compound **6** (crystallographic numbering)

the case of hydroxamates, it is probable that the equilibrium is shifted toward the cuprate **8**, because hydroxamates are more acidic than the corresponding amides,[33] and, as a result, the concentration of the amide anion is higher. Cross-coupling with the aryl iodide proceeds after dissociation of cuprate **8** to the copper amidate **7**. However, in the case of α-bromo derivative **1w**, it is difficult to decide

whether the arylation proceeds via the cuprate **8**, the amidate **7**, or some other species, but two molecules of the hydroxamate **1w** have to interact during the course of reaction.



Scheme 2

Our hypothesis regarding the presence of cuprate complex **8** is indirectly supported by another observation. The yields of *N*-aryl-*O*-allylhydroxamates were higher than those of the corresponding *O*-methyl derivatives, especially if less reactive aryl iodides were used (Table 1, entries 12 and 13; Table 2, entries 23 and 24, 26 and 27, and 28 and 29). We assume that the double bond of the allyl group can coordinate to copper,[34] thereby sterically protecting the amidate from addition of the second amide anion and thereby shifting the equilibrium from **8** toward **7**.

In summary, we have demonstrated the scope and limitations of the copper-catalyzed N-arylation of hydroxamates with aryl and hetaryl iodides. The method is applicable, with some exceptions, to various types of hydroxamic acid derivatives and it tolerates various functional groups that are suitable for further modification.

Solvents and reagents were purchased from commercial suppliers and were used without additional purification. Flash chromatography was carried out by using Merck Kieselgel 60 H silica. Analytical TLC was carried out on silica gel 60 F254 plates (0.2 mm, Merck), which were visualized under UV radiation (at 254 and/or 365 nm). GC analyses of the crude reaction mixtures and pure products were performed on an Agilent Technologies 7890A GC system with the inert XL EI/CI 5975C triple-axis detector. NMR spectra were recorded on a Varian Mercury 400 spectrometer. Chemical shifts (δ) are reported in ppm relative to the solvent peak (DMSO-d_6 or $CDCl_3$) as a reference, and coupling constants *J* are reported in Hz. High-resolution mass spectra were recorded in the positive electrospray ionization (ESI^+) mode on a Micromass Q-Tof micro instrument. IR spectra were recorded on a Shimadzu IR Prestige-21 spectrometer. Melting points were recorded with an Optimelt apparatus and are uncorrected. Elemental analyses were performed on Carlo-Erba EA-1108 instrument. X-ray crystal structures were recorded on Bruker–Nonius Kappa CCD diffractometer.

Analytical data for compounds **4** and **5** have been reported elsewhere.[30]

Crystallographic data for compounds **2e**, **3h**, and **6** have been deposited at the Cambridge Crystallographic Data Centre with the accession numbers CCDC 868894, 868893, and 868895, respectively, and can be obtained free of charge from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44(1223)336033; E-mail: deposit@ccdc.cam.ac.uk; Web site: www.ccdc.cam.ac.uk/conts/retrieving.html.

N-Arylation of Hydroxamate Derivatives with Aryl Iodides; General Procedure

An oven-dried vial equipped with a stirrer bar was charged with the hydroxamate derivative (0.20 mmol), Cu_2O (2.9 mg, 0.020 mmol), K_2CO_3 (55 mg, 0.40 mmol), aryl iodide (0.30 mmol, if solid at r.t.), and 3 Å MS (100% of weight). The vial was closed with an open-top aluminum seal with a PTFE-faced septum and flushed with argon. Anhyd toluene (2 mL), DMEDA (4.3 μL, 0.040 mmol), and the aryl iodide (0.30 mmol, if liquid at r.t.) were added, and the mixture was stirred at 80 °C for 18 h. The mixture was cooled, diluted with EtOAc (5 mL), filtered through a short plug of silica gel, and washed with EtOAc. The solvent was removed in vacuo and the crude product was purified by flash column chromatography [silica gel, EtOAc–PE (1:5)] to give the desired product (Tables 1 and 2).

***N*-Methoxy-*N*,*2*-diphenylacetamide (2a)[35]**

White powder; yield: 43 mg (88%) (0.2 mmol scale); 218 mg (90%) (1.0 mmol scale).

IR (film): 3030, 2937, 1675, 1594, 1493, 1362, 1277, 1065, 979, 756, 726, 695 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.49–7.40 (m, 4 H), 7.36–7.22 (m, 6 H), 3.91 (s, 2 H), 3.68 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.2, 138.1, 134.6, 129.4, 128.9, 128.6, 126.9, 123.2, 61.8, 40.9.

Anal. Calcd for $C_{15}H_{15}NO_2$: C, 74.67; H, 6.27; N, 5.80. Found: C, 74.40; H, 6.25; N, 5.70.

***N*-(Allyloxy)-*N*,*2*-diphenylacetamide (2b)[36]**

Yellow oil; yield: 51 mg (96%).

IR (film): 3030, 2928, 1680, 1493, 1361, 757, 694 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.48–7.40 (m, 4 H), 7.35–7.22 (m, 6 H), 6.00 (ddt, *J* = 17.2, 10.3, 6.3 Hz, 1 H), 5.39 (dd, *J* = 17.2, 1.5 Hz, 1 H), 5.33 (dd, *J* = 10.3, 1.5 Hz, 1 H), 4.37 (d, *J* = 6.3 Hz, 2 H), 3.91 (s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 138.6, 134.6, 131.1, 129.4, 128.9, 128.5, 126.9, 120.9, 75.1, 41.0.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{17}H_{17}NO_2$: 268.1338; found: 268.1358.

***N*-(Benzyloxy)-*N*,*2*-diphenylacetamide (2c)[36]**

Yellow oil; yield: 45 mg (71%).

IR (film): 3031, 1671, 1491, 1363, 759, 694 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.54–7.39 (m, 9 H), 7.33–7.26 (m, 3 H), 7.25–7.14 (m, 3 H), 4.89 (s, 2 H), 3.85 (s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 134.6, 134.2, 129.6, 129.4, 129.1, 128.9, 128.7, 128.5, 126.9, 76.2, 40.8.

Anal. Calcd for $C_{21}H_{19}NO_2$: C, 79.47; H, 6.03; N, 4.41. Found: C, 79.15; H, 5.96; N, 4.31.

***N*,2-Diphenyl-*N*-(tetrahydro-2*H*-pyran-2-yloxy)acetamide (2d)[36]**

Colorless oil; yield: 50 mg (81%).

IR (film): 2947, 2854, 1683, 1492, 1356, 1034, 696 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.45–7.37 (m, 4 H), 7.34–7.18 (m, 6 H), 5.05–4.98 (m, 1 H), 3.87 (s, 2 H), 3.70–3.59 (m, 1 H), 3.38–3.32 (m, 1 H), 1.79–1.61 (m, 3 H), 1.58–1.41 (m, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 140.2, 134.7, 129.4, 128.9, 128.4, 126.7, 102.3, 63.3, 40.8, 28.6, 25.0, 19.3.

Anal. Calcd for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80; N, 4.50. Found: C, 73.29; H, 6.84; N, 4.42.

***N*-(*tert*-Butoxy)-*N*,2-diphenylacetamide (2e)[36]**

White solid; yield: 41 mg (73%); mp 78–80 °C.

IR (film): 2979, 1685, 1491, 1367, 1180, 695 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.42–7.35 (m, 4 H), 7.32–7.16 (m, 6 H), 3.86 (s, 2 H), 1.20 (s, 9 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.4, 134.8, 129.4, 128.5, 128.4, 126.8, 126.7, 125.1, 84.3, 40.6, 27.8.

Anal. Calcd for $C_{18}H_{21}NO_2$: C, 76.30; H, 7.47; N, 4.94. Found: C, 75.90; H, 7.39; N, 4.91.

***N*-Methoxy-*N*,2-diphenylpropanamide (2g)[36]**
Colorless oil; yield: 42 mg (82%).

IR (film): 2974, 2934, 1678, 1593, 1489, 1454, 1375, 1360, 1275, 1057, 754, 696, 546 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.50–7.15 (m, 10 H), 4.46–3.93 (m, 1 H), 3.46 (s, 3 H), 1.38 (d, *J* = 7.0 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 173.3, 141.7, 138.2, 128.8, 128.7, 127.7, 126.9, 123.0, 61.6, 43.5, 19.8.

Anal. Calcd for $C_{16}H_{17}NO_2$: C, 75.27; H, 6.71; N, 5.49. Found: C, 75.02; H, 6.65; N, 5.39.

***N*-Methoxy-*N*,1-diphenylcyclopropanecarboxamide (2h)**
Colorless oil; yield: 37 mg (70%).

IR (film): 3060, 3009, 2936, 1674, 1668, 1495, 1489, 1350, 759, 695, 537 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.39–7.33 (m, 2 H), 7.31–7.23 (m, 5 H), 7.22–7.15 (m, 3 H), 3.20 (s, 3 H), 1.45–1.40 (m, 2 H), 1.16–1.11 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 172.1, 140.6, 138.8, 128.7, 128.3, 127.7, 126.9, 126.4, 124.5, 60.6, 30.8, 14.1.

Anal. Calcd for $C_{17}H_{17}NO_2$: C, 76.38; H, 6.41; N, 5.24. Found: C, 76.18; H, 6.40; N, 5.22.

***N*-Methoxy-*N*-phenylcyclohexanecarboxamide (2i)[36]**
Yellow oil; yield: 29 mg (63%) (0.2 mmol scale); 155 mg (66%) (1.0 mmol scale).

IR (film): 2931, 2855, 1679, 1492, 1379, 1341, 1273, 755, 692 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53–7.42 (m, 2 H), 7.42–7.35 (m, 2 H), 7.28–7.20 (m, 1 H, overlapped by $CDCl_3$), 3.71 (s, 3 H), 2.90–2.51 (m, 1 H), 1.97–1.74 (m, 4 H), 1.73–1.63 (m, 1 H), 1.62–1.48 (m, 2 H), 1.39–1.17 (m, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 138.6, 128.8, 126.6, 123.5, 62.0, 41.3, 29.1, 25.8, 25.7.

HRMS (ESI): *m/z* [M + Na]$^+$ calcd for $C_{14}H_{19}NO_2$ + Na: 256.1313; found: 256.1311.

***N*-Methoxy-*N*-phenylpentanamide (2j)[36,37]**
Yellow oil; yield: 39 mg (95%).

IR (film): 2959, 2935, 2873, 1682, 1594, 1493, 1379, 1279, 1066, 980, 755, 693 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.51–7.44 (m, 2 H), 7.42–7.36 (m, 2 H), 7.26–7.20 (m, 1 H, overlapped by $CDCl_3$), 3.71 (s, 3 H), 2.63–2.44 (m, 2 H), 1.74–1.63 (m, 2 H), 1.46–1.34 (m, 2 H), 0.94 (t, *J* = 7.4 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 172.5, 138.4, 128.8, 126.4, 123.1, 61.7, 33.1, 26.7, 22.4, 13.8.

HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{12}H_{18}NO_2$: 208.1338; found: 208.1347.

***N*-Methoxy-*N*-phenyltetrahydrofuran-2-carboxamide (2k)[36]**
Yellow oil; yield: 18 mg (41%).

IR (film): 2973, 2875, 1695, 1593, 1489, 1382, 1287, 1079, 1062, 758, 693 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.56–7.46 (m, 2 H), 7.44–7.35 (m, 2 H), 7.28–7.19 (m, 1 H, overlapped by $CDCl_3$), 5.03–4.69 (m, 1 H), 4.08 (q, *J* = 7.0 Hz, 1 H), 4.01–3.92 (m, 1 H), 3.73 (s, 3 H), 2.36–2.18 (m, 1 H), 2.17–2.07 (m, 1 H), 2.07–1.98 (m, 1 H), 1.97–1.85 (m, 1 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.6, 137.8, 128.9, 126.6, 122.3, 76.0, 69.6, 61.9, 29.7, 25.5.

HRMS (ESI): *m/z* [M + Na]$^+$ calcd for $C_{12}H_{15}NO_3$ + Na: 244.0950; found: 244.0966.

***N*-Methoxy-*N*-phenylbenzamide (2l)**
Light-yellow oil; yield: 16 mg (35%).

IR (film): 3061, 1666, 1489, 1352, 694 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.64–7.59 (m, 2 H), 7.47–7.32 (m, 7 H), 7.28–7.22 (m, 1 H, overlapped by $CDCl_3$), 3.70 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.1, 139.2, 134.6, 130.6, 128.9, 128.4, 128.0, 127.0, 124.4, 61.6.

Anal. Calcd for $C_{14}H_{13}NO_2$: C, 73.99; H, 5.77; N, 6.16. Found: C, 73.82; H, 5.72; N, 6.01.

***N*-(Allyloxy)-*N*-phenylbenzamide (2m)**
Light-yellow oil; yield: 22 mg (44%)

IR (film): 3063, 1665, 1490, 1352, 1306, 761, 694 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.64–7.59 (m, 2 H), 7.49–7.44 (m, 2 H), 7.43–7.30 (m, 5 H), 7.26–7.20 (m, 1 H, overlapped by $CDCl_3$), 5.80 (ddt, *J* = 16.8, 10.2, 6.3 Hz, 1 H), 5.28–5.19 (m, 2 H), 4.36 (d, *J* = 6.3 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 168.4, 139.8, 134.8, 131.4, 130.6, 129.0, 128.7, 127.9, 127.0, 124.4, 120.8, 75.3.

HRMS (ESI): *m/z* [M + Na]$^+$ calcd for $C_{16}H_{15}NO_2$ + Na: 276.1000; found: 276.0978.

***N*,4-Dimethoxy-*N*-phenylbenzamide (2n)**
Colorless oil; yield: 14 mg (26%) (0.2 mmol scale); 52 mg (20%) (1.0 mmol scale).

IR (film): 3065, 2969, 2839, 1654, 1649, 1607, 1490, 1348, 1305, 1256, 1176, 1030, 840, 753, 695, 609 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.67–7.60 (m, 2 H), 7.45–7.40 (m, 2 H), 7.39–7.32 (m, 2 H), 7.26–7.20 (m, 1 H, overlapped by $CDCl_3$), 6.87–6.81 (m, 2 H), 3.82 (s, 3 H), 3.72 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 167.6, 161.6, 139.8, 130.8, 129.9, 126.9, 126.5, 124.5, 113.3, 61.6, 55.3.

HRMS (ESI): *m/z* [M + Na]$^+$ calcd for $C_{15}H_{15}NO_3$ + Na: 280.0950; found: 280.0967.

***N*-Methoxy-*N*-phenyl-4-(trifluoromethyl)benzamide (2o)**
Colorless oil; yield: 36 mg (60%).

IR (film): 3067, 2938, 1668, 1492, 1361, 1332, 1318, 1169, 1128, 1067, 1017, 860, 841, 760, 693 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.78–7.71 (m, 2 H), 7.67–7.60 (m, 2 H), 7.51–7.45 (m, 2 H), 7.43–7.37 (m, 2 H), 7.32–7.26 (m, 1 H), 3.67 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 166.8, 138.5, 138.1, 132.7 (q, $^2J_{C,F}$ = 31.5 Hz), 129.1, 128.7, 127.4, 125.1 (d, $^3J_{C,F}$ = 3.2 Hz), 124.1, 122.3, 61.8.

HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{15}H_{13}{}^{19}F_3NO_2$: 296.0898; found: 296.0892.

***N*-Methoxy-*N*-phenylthiophene-3-carboxamide (2p)**
Colorless oil; yield: 30 mg (64%).

IR (film): 3108, 2934, 1638, 1592, 1051, 970, 838 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.18–8.14 (m, 1 H), 7.60–7.57 (m, 1 H), 7.54–7.49 (m, 2 H), 7.49–7.43 (m, 2 H), 7.39 (dd, *J* = 5.1, 1.2 Hz, 1 H), 7.35–7.30 (m, 1 H), 3.68 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 162.2, 139.0, 135.1, 131.3, 129.02, 128.99, 127.1, 124.9, 124.2, 61.8.

Anal. Calcd for $C_{12}H_{11}NO_2S$: C, 61.78; H, 4.75; N, 6.00. Found: C, 61.39; H, 4.55; N, 5.88.

***N*-Methoxy-*N*-phenylfuran-2-carboxamide (2q)**
Colorless oil; yield: 22 mg (51%).

IR (film): 3300, 3119, 2636, 1651, 1468, 1393, 1358, 1022, 758, 694 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.63–7.56 (m, 3 H), 7.47–7.40 (m, 2 H), 7.32–7.26 (m, 1 H), 7.16–7.12 (m, 1 H), 6.54–6.51 (m, 1 H), 3.79 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.3, 146.0, 145.8, 138.4, 129.1, 127.1, 123.6, 118.1, 111.8, 62.1.

Anal. Calcd for $C_{12}H_{11}NO_3$: C, 66.35; H, 5.10; N, 6.45. Found: C, 66.42; H, 5.04; N, 6.40.

***N*-Methoxy-*N*-phenylfuran-3-carboxamide (2r)**
Colorless oil; yield: 31 mg (72%).

IR (film): 1653, 1490, 1388, 1350, 876, 757, 739, 694, 602 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.35 (s, 1 H), 7.83–7.79 (m, 1 H), 7.58–7.53 (m, 2 H), 7.51–7.44 (m, 2 H), 7.35–7.29 (m, 1 H), 6.83 (d, *J* = 1.2 Hz, 1 H), 3.71 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 161.4, 146.8, 142.7, 138.4, 129.0, 126.9, 123.5, 120.5, 111.6, 61.8.

Anal. Calcd for $C_{12}H_{11}NO_3$: C, 66.35; H, 5.10; N, 6.45. Found: C, 66.00; H, 5.01; N, 6.32.

3-(2-Bromophenyl)-*N*-methoxy-*N*-phenylpropanamide (2s)[36]
Yellow oil; yield: 52 mg (82%).

IR (film): 3064, 2936, 1681, 1493, 1376, 1025, 754, 693 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54 (d, *J* = 7.8 Hz, 1 H), 7.48–7.30 (m, 5 H), 7.27–7.20 (m, 2 H, overlapped by $CDCl_3$), 7.09 (dt, *J* = 7.8, 1.6 Hz, 1 H), 3.65 (s, 3 H), 3.15 (t, *J* = 7.8 Hz, 2 H), 3.00–2.75 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 140.4, 138.1, 132.9, 131.0, 128.9, 128.0, 127.6, 124.5, 61.8, 33.4, 31.4.

Anal. Calcd for $C_{16}H_{16}BrNO_2$: C, 57.50; H, 4.83; N, 4.19. Found: C, 57.68; H, 4.81; N, 3.98.

3-(2-Iodophenyl)-*N*-methoxy-*N*-phenylpropanamide (2t)[36]
Yellow oil; yield: 8 mg (10%).

IR (film): 1680, 1492, 1347, 1011, 753 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (d, *J* = 7.8 Hz, 1 H), 7.56–7.35 (m, 4 H), 7.33 (dd, *J* = 7.8, 1.6 Hz, 1 H), 7.28 (dt, *J* = 7.8, 1.6 Hz, 1 H, overlapped by $CDCl_3$), 7.26–7.20 (m, 1 H, overlapped by $CDCl_3$), 6.91 (dt, *J* = 7.8, 1.6 Hz, 1 H), 3.65 (s, 3 H), 3.13 (t, *J* = 7.8 Hz, 2 H), 3.00–2.75 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.6, 139.5, 138.1, 130.0, 128.9, 128.5, 128.1, 126.6, 123.1, 100.3, 61.8, 35.8, 33.6.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{16}H_{17}NO_2{}^{127}I$: 382.0304; found: 382.0320.

2-(2-Chlorophenyl)-*N*-methoxy-*N*-phenylacetamide (2u)[36]
Colorless oil; yield: 37 mg (67%).

IR (film): 3334, 3063, 2937, 1672, 1594, 1491, 1364, 1359, 1269, 1065, 979, 749, 692, 555 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.55–7.48 (m, 2 H), 7.42–7.32 (m, 4 H), 7.27–7.19 (m, 3 H, overlapped by $CDCl_3$), 4.05 (s, 2 H), 3.72 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 169.3, 138.0, 134.5, 133.0, 131.5, 129.5, 128.6, 128.5, 126.9, 122.8, 61.9, 38.5.

Anal. Calcd for $C_{15}H_{14}ClNO_2$: C, 65.34; H, 5.12; N, 5.05. Found: C, 65.20; H, 4.94; N, 5.01.

Ethyl 4-[Methoxy(phenylacetyl)amino]benzoate (3a)
Colorless oil; yield: 57 mg (90%).

IR (film): 2980, 1714, 1688, 1605, 1505, 1350, 1275, 1175, 1107, 1071, 856, 770, 729, 697 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.04–7.92 (m, 2 H), 7.70–7.64 (m, 2 H), 7.38–7.22 (m, 5 H), 4.31 (q, *J* = 7.0 Hz, 2 H), 4.01 (s, 2 H), 3.76 (s, 3 H), 1.32 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.8, 166.0, 141.9, 134.2, 130.3, 129.5, 128.7, 127.5, 127.1, 120.8, 62.3, 61.0, 41.1, 14.4.

Anal. Calcd for $C_{18}H_{19}NO_4$: C, 69.00; H, 6.11; N, 4.47. Found: C, 68.66; H, 6.10; N, 4.42.

Ethyl 3-[Methoxy(phenylacetyl)amino]benzoate (3b)
Colorless oil; yield: 58 mg (92%).

IR (film): 2980, 1719, 1684, 1586, 1445, 1367, 1285, 1107, 757, 704 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 8.08–8.04 (m, 1 H), 7.83 (d, *J* = 8.2 Hz, 1 H), 7.76–7.71 (m, 1 H), 7.59 (t, *J* = 8.2 Hz, 1 H), 7.37–7.22 (m, 5 H), 4.33 (q, *J* = 7.0 Hz, 2 H), 3.98 (s, 2 H), 3.72 (s, 3 H), 1.32 (t, *J* = 7.0 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.7, 166.0, 138.3, 134.4, 131.4, 129.4, 128.8, 128.6, 127.5, 127.0, 123.4, 62.1, 61.3, 40.9, 14.3.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{18}H_{20}NO_4$: 314.1392; found: 314.1410.

***N*-Methoxy-*N*-(4-nitrophenyl)-2-phenylacetamide (3c1)**
Orange solid; yield: 53 mg (92%); mp 73–74 °C.

IR (film): 1694, 1592, 1517, 1491, 1341, 1274, 1112, 854, 750, 731, 694 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.28–8.21 (m, 2 H), 7.80–7.74 (m, 2 H), 7.40–7.27 (m, 5 H), 4.01 (s, 2 H), 3.69 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.3, 144.5, 143.6, 133.7, 129.5, 128.8, 127.3, 124.6, 120.4, 62.7, 41.1.

HRMS (ESI): *m/z* $[M + Na]^+$ calcd for $C_{15}H_{14}N_2O_4$ + Na: 309.0851; found: 309.0860.

***N*-(Allyloxy)-*N*-(4-nitrophenyl)-2-phenylacetamide (3c2)**
Yellow solid; yield: 56 mg (90%); mp 68–69 °C.

IR (film): 3030, 2931, 1692, 1592, 1517, 1341, 1274, 1178, 1112, 942, 853, 751 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.27–8.21 (m, 2 H), 7.80–7.73 (m, 2 H), 7.40–7.27 (m, 5 H), 6.01–5.89 (m, 1 H), 5.44–5.36 (m, 2 H), 4.29 (d, *J* = 6.3 Hz, 2 H), 4.02 (s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.6, 144.6, 144.2, 133.7, 130.1, 129.5, 128.7, 127.3, 124.5, 121.9, 120.8, 76.2, 41.2.

HRMS (ESI): *m/z* $[M + Na]^+$ calcd for $C_{17}H_{16}N_2O_4$ + Na: 335.1008; found: 335.0987.

***N*-Methoxy-*N*-(3-nitrophenyl)-2-phenylacetamide (3d)**
Yellow oil; yield: 47 mg (82%).

IR (film): 1687, 1528, 1348, 737, 703 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.42–8.39 (m, 1 H), 8.05 (dd, *J* = 8.2, 2.0 Hz, 1 H), 7.96–7.92 (m, 1 H), 7.54 (t, *J* = 8.2 Hz, 1 H), 7.40–7.27 (m, 5 H), 4.00 (s, 2 H), 3.67 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.3, 148.6, 139.4, 133.8, 129.6, 129.4, 128.7, 127.4, 127.3, 120.4, 116.0, 62.6, 40.9.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{15}H_{15}N_2O_4$: 287.1032; found: 287.1040.

***N*-(4-Acetylphenyl)-*N*-methoxy-2-phenylacetamide (3e1)**
Colorless oil; yield: 52 mg (89%).

IR (film): 3341, 1683, 1599, 1499, 1359, 1267, 1179, 839, 708 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.01–7.95 (m, 2 H), 7.69–7.63 (m, 2 H), 7.39–7.27 (m, 5 H), 3.98 (s, 2 H), 3.65 (s, 3 H), 2.59 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 197.0, 170.9, 142.2, 134.2, 134.1, 129.4, 129.2, 128.7, 127.1, 120.8, 62.4, 41.1, 26.6.

HRMS (ESI): *m/z* $[M + Na]^+$ calcd for $C_{17}H_{17}NO_3$ + Na: 306.1106; found: 306.1133.

***N*-(4-Acetylphenyl)-*N*-(allyloxy)-2-phenylacetamide (3e2)**
Yellow oil; yield: 51 mg (84%) (0.2 mmol scale); 280 mg (90%) (1.0 mmol scale).

IR (film): 3030, 1681, 1599, 1499, 1416, 1359, 1267, 1179, 959, 839, 707 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 8.00–7.94 (m, 2 H), 7.69–7.63 (m, 2 H), 7.38–7.26 (m, 5 H), 6.02–5.87 (m, 1 H), 5.41–5.32 (m, 2 H), 4.26 (d, *J* = 6.3 Hz, 2 H), 3.99 (s, 2 H), 2.59 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 197.0, 171.2, 142.7, 134.3, 134.1, 130.5, 129.5, 129.2, 128.6, 127.1, 121.5, 121.1, 75.7, 41.2, 26.6.

HRMS (ESI): *m/z* $[M + Na]^+$ calcd for $C_{19}H_{19}NO_3$ + Na: 332.1263; found: 332.1286.

***N*-(Allyloxy)-*N*-(4-cyanophenyl)-2-phenylacetamide (3f)**
Colorless oil; yield: 56 mg (90%).

IR (film): 2227, 1689, 1602, 1497, 1348, 1277, 1176, 840, 711 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.73–7.68 (m, 2 H), 7.67–7.63 (m, 2 H), 7.38–7.27 (m, 5 H), 6.00–5.87 (m, 1 H), 5.43–5.33 (m, 2 H), 4.27 (d, *J* = 6.3 Hz, 2 H), 3.99 (s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.4, 142.5, 133.8, 132.9, 130.2, 129.4, 128.7, 127.3, 121.8, 121.3, 118.6, 108.8, 76.0, 41.1.

HRMS (ESI): *m/z* $[M + Na]^+$ calcd for $C_{18}H_{16}N_2O_2$ + Na: 315.1109; found: 315.1108.

***N*-(Allyloxy)-*N*-(3,4-difluorophenyl)-2-phenylacetamide (3g)[36]**
Colorless oil; yield: 56 mg (93%).

IR (film): 3031, 1684, 1611, 1511, 1425, 1360, 1261, 1209, 966, 939, 866, 816, 778, 720 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.61–7.46 (m, 2 H), 7.36–7.22 (m, 6 H), 6.02 (ddt, *J* = 17.2, 10.4, 6.3 Hz, 1 H), 5.42 (dd, *J* = 17.2, 1.5 Hz, 1 H), 5.34 (dd, *J* = 10.4, 1.5 Hz, 1 H), 4.42 (d, *J* = 6.3 Hz, 2 H), 3.94 (s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.1, 151.3 (d, $^2J_{C,F}$ = 13.6 Hz), 148.8 (d, $^2J_{C,F}$ = 13.6 Hz), 135.0, 134.2, 130.6, 129.4, 128.6, 127.1, 121.4, 118.8, 117.1 (d, $^2J_{C,F}$ = 13.6 Hz), 112.3, 75.6, 40.9.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{17}H_{16}{}^{19}F_2NO_2$: 304.1149; found: 304.1144.

***N*-Methoxy-*N*-(3-methoxyphenyl)-2-phenylacetamide (3h)[36]**
Yellow solid; yield: 53 mg (97%); mp 56–58 °C.

IR (film): 2938, 1679, 1603, 1487, 1358, 1043, 778, 719, 695 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.37–7.21 (m, 6 H), 7.07–7.02 (m, 2 H), 6.85 (dd, *J* = 8.2, 1.8 Hz, 1 H), 3.90 (s, 2 H), 3.75 (s, 3 H), 3.69 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.2, 160.0, 139.2, 134.6, 129.5, 129.4, 128.6, 126.9, 115.3, 112.6, 108.4, 62.0, 55.4, 41.0.

HRMS (ESI): *m/z* $[M + Na]^+$ calcd for $C_{16}H_{17}NO_3$ + Na: 294.1106; found: 294.1093.

***N*-Methoxy-2-phenyl-*N*-(4-tolyl)acetamide (3j1)[36]**
Colorless oil; yield: 36 mg (71%).

IR (film): 3030, 2935, 1676, 1507, 1362, 1278, 1069, 980, 818, 717 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.39–7.17 (m, 9 H), 4.0–3.8 (br s, 2 H), 3.64 (s, 3 H), 2.31 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 134.7, 129.5, 129.3, 128.5, 126.8, 61.6, 40.8, 21.1.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{16}H_{18}NO_2$: 256.1338; found: 256.1352.

***N*-(Allyloxy)-2-phenyl-*N*-(4-tolyl)acetamide (3j2)[36]**
Yellow oil; yield: 45 mg (79%).

IR (film): 3030, 2923, 1674, 1506, 1362, 818, 720, 696 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.36–7.20 (m, 9 H), 6.01 (ddt, *J* = 17.2, 10.6, 6.3 Hz, 1 H), 5.37 (dd, *J* = 17.2, 1.2 Hz, 1 H), 5.31 (dd, *J* = 10.6, 1.2 Hz, 1 H), 4.34 (d, *J* = 6.3 Hz, 2 H), 3.9–3.7 (br s, 2 H), 2.31 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 134.8, 131.4, 129.5, 129.4, 128.5, 126.8, 120.8, 74.9, 40.9, 21.1.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{18}H_{20}NO_2$: 282.1494; found: 282.1523.

***N*-(Benzyloxy)-2-phenyl-*N*-(4-tolyl)acetamide (3j3)[36]**
Colorless oil; yield: 52 mg (79%).

IR (film): 3063, 3031, 2922, 1674, 1506, 1362, 1277, 818, 754, 719, 700 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 7.44–7.27 (m, 9 H), 7.26–7.17 (m, 5 H), 4.76 (s, 2 H), 3.77 (s, 2 H), 2.38 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.6, 135.9, 134.7, 134.3, 129.5, 129.3, 128.9, 128.6, 128.4, 126.8, 123.7, 75.9, 40.6, 21.1.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{22}H_{22}NO_2$: 332.1651; found: 332.1661.

***N*-Methoxy-*N*-(4-methoxyphenyl)-2-phenylacetamide (3k)[36]**
Colorless oil; yield: 39 mg (72%) (0.2 mmol scale); 239 mg (87%) (1.0 mmol).

IR (film): 1674, 1506, 1368, 1249, 1031, 832 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.37–7.18 (m, 7 H), 7.02–6.95 (m, 2 H), 4.0–3.7 (br s, 2 H), 3.77 (s, 3 H), 3.62 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 134.8, 129.3, 128.5, 126.8, 114.2, 61.4, 55.5, 40.8.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{16}H_{18}NO_3$: 272.1287; found: 272.1276.

***N*-(4-Bromophenyl)-*N*-methoxy-2-phenylacetamide (3l)[36]**
Yellow oil; yield: 48 mg (75%).

IR (film): 3030, 2936, 1684, 1484, 1356, 1275, 1070, 978, 826, 730, 698 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.65–7.58 (m, 2 H), 7.48–7.41 (m, 2 H), 7.37–7.21 (m, 5 H), 3.94 (s, 2 H), 3.70 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.5, 137.2, 134.3, 131.9, 129.4, 128.6, 127.0, 124.1, 62.1, 40.9.

HRMS (ESI): *m/z* $[M + H]^+$ calcd for $C_{15}H_{15}{}^{79}BrNO_2$: 320.0286; found: 320.0292.

***N*-(4-Chlorophenyl)-*N*-methoxy-2-phenylacetamide (3m)[36]**
Yellow oil; yield: 53 mg (82%).

IR (film): 2937, 1895, 1682, 1488, 1356, 1275, 1094, 1069, 979, 829, 736 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 7.55–7.45 (m, 4 H), 7.37–7.21 (m, 5 H), 3.94 (s, 2 H), 3.70 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.5, 136.6, 134.4, 129.4, 128.9, 128.6, 127.0, 123.9, 62.0, 40.8.

HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{15}H_{15}{}^{35}ClNO_2$: 276.0791; found: 276.0775.

Methyl 3-Chloro-5-[methoxy(phenylacetyl)amino]benzoate (3n)
Colorless oil; yield: 58 mg (88%).

IR (film): 2951, 1729, 1688, 1582, 1453, 1433, 1349, 1294, 974, 768, 702 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.08–8.05 (m, 1 H), 7.86–7.84 (m, 1 H), 7.81–7.78 (m, 1 H), 7.39–7.35 (m, 1 H), 7.35–7.27 (m, 4 H), 3.96 (s, 2 H), 3.92 (s, 3 H), 3.64 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.0, 165.4, 139.5, 134.8, 134.0, 132.2, 129.4, 128.7, 127.2, 127.0, 126.0, 120.6, 62.5, 52.6, 40.1.

HRMS (ESI): *m/z* [M + Na]$^+$ calcd for $C_{17}H_{16}{}^{35}ClNO_4$ + Na: 356.0666; found: 356.0674.

***N*-Methoxy-2-phenyl-*N*-pyridin-3-ylacetamide (3o)[36]**
Brown solid; mp 76–78 °C; yield: 43 mg (89%) (0.2 mmol); 214 mg (88%) (1.0 mmol scale).

IR (film): 3030, 2938, 1684, 1477, 1360, 1316, 1282, 1077, 976, 805, 731, 701 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.27–8.28 (m, 2 H), 7.90 (d, *J* = 7.8 Hz, 1 H), 7.44–7.26 (m, 6 H), 3.96 (s, 2 H), 3.65 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.1, 147.0, 143.1, 134.0, 129.4, 129.2, 128.6, 127.1, 123.9, 62.4, 40.7.

HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{14}H_{15}N_2O_2$: 243.1134; found: 243.1151.

***N*-(5-Bromopyridin-2-yl)-*N*-methoxy-2-phenylacetamide (3p)**
Orange oil; yield: 40 mg (63%).

IR (film): 3030, 2936, 1690, 1686, 1570, 1456, 1350, 1271, 1096, 736, 704 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.52 (d, *J* = 2.3 Hz, 1 H), 7.80 (dd, *J* = 8.6, 2.3 Hz, 1 H), 7.73 (d, *J* = 8.6 Hz, 1 H), 7.39–7.26 (m, 5 H), 4.03 (s, 2 H), 3.79 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.2, 149.7, 148.9, 140.4, 133.9, 129.5, 128.6, 127.1, 118.4, 116.7, 63.5, 41.4.

HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{14}H_{14}{}^{79}BrN_2O_2$: 321.0239; found: 321.0232.

***N*-(2-Fluorophenyl)-*N*-methoxy-2-phenylacetamide (3q1)[36]**
Colorless oil; yield: 20 mg (38%).

IR (film): 3030, 2936, 1685, 1591, 1497, 1454, 1369, 1266, 759, 725 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.56–7.43 (m, 2 H), 7.42–7.18 (m, 7 H), 4.1–3.7 (br s, 2 H), 3.65 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.9 (d, $^1J_{C,F}$ = 253.8 Hz), 134.4 (d, $^3J_{C,F}$ = 7.4 Hz), 130.6 (d, $^3J_{C,F}$ = 6.2 Hz), 129.3, 128.5, 126.9, 124.6 (d, $^4J_{C,F}$ = 2.3 Hz), 116.6 (d, $^2J_{C,F}$ = 20.2 Hz), 61.9, 40.4.

HRMS (ESI): *m/z* [M + Na]$^+$ calcd for $C_{15}H_{14}{}^{19}FNO_2$ + Na: 282.0906; found: 282.0919.

***N*-(Allyloxy)-*N*-(2-fluorophenyl)-2-phenylacetamide (3q2)[36]**
Colorless oil; yield: 36 mg (63%).

IR (film): 3030, 2928, 1684, 1611, 1497, 1454, 1368, 759 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.53–7.43 (m, 2 H), 7.39–7.18 (m, 7 H), 5.93 (ddt, *J* = 17.2, 10.4, 6.3 Hz, 1 H), 5.35 (dd, *J* = 17.2, 1.2 Hz, 1 H), 5.28 (dd, *J* = 10.4, 1.2 Hz, 1 H), 4.37 (d, *J* = 6.3 Hz, 2 H), 4.1–3.7 (br s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.8 (d, $^1J_{C,F}$ = 253.8 Hz), 134.4, 131.3, 130.5, 129.3, 128.5, 126.9, 124.6 (d, $^4J_{C,F}$ = 3.5 Hz), 120.9, 116.6 (d, $^2J_{C,F}$ = 20.2 Hz), 75.6, 40.4.

HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{17}H_{17}{}^{19}FNO_2$: 286.1243; found: 286.1242.

***N*-(Benzyloxy)-*N*-(2-fluorophenyl)-2-phenylacetamide (3q3)[36]**
Light-yellow oil; yield: 32 mg (48%) (0.2 mmol scale); 135 mg (40%) (1.0 mmol scale).

IR (film): 3064, 3031, 1684, 1496, 1369, 758, 727, 697 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.53–7.43 (m, 2 H), 7.41–7.11 (m, 12 H), 4.90 (s, 2 H), 3.9–3.7 (br s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.1, 157.7 (d, $^1J_{C,F}$ = 253.8 Hz), 130.4, 134.3, 129.5, 129.3, 128.9, 128.5, 128.5, 126.9, 124.6 (d, $^4J_{C,F}$ = 3.9 Hz), 116.6 (d, $^2J_{C,F}$ = 20.2 Hz), 76.7, 40.2.

HRMS (ESI): *m/z* [M + Na]$^+$ calcd for $C_{21}H_{18}{}^{19}FNO_2$ + Na: 358.1219; found: 358.1219.

***N*-Methoxy-*N*-(2-methylphenyl)-2-phenylacetamide (3r1)[36]**
Yellow oil; yield: 12 mg (24%).

IR (film): 3029, 1670, 1491, 1374, 1369, 721 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.49–7.18 (m, 9 H), 4.2–3.7 (br s, 2 H), 3.58 (s, 3 H), 2.10 (s, 3 H).

^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.3, 136.9, 136.3, 135.1, 130.7, 129.4, 129.0, 128.8, 128.2, 127.6, 126.5, 61.3, 17.4.

HRMS (ESI): *m/z* [M + Na]$^+$ calcd for $C_{16}H_{17}NO_2$ + Na: 278.1157; found: 278.1176.

***N*-(Allyloxy)-*N*-(2-methylphenyl)-2-phenylacetamide (3r2)[36]**
Yellow oil; yield: 20 mg (35%).

IR (film): 3029, 1676, 1490, 1369, 1364, 723 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.46–7.15 (m, 9 H), 5.92 (ddt, *J* = 17.2, 10.6, 6.3 Hz, 1 H), 5.33 (d, *J* = 17.2 Hz, 1 H), 5.26 (d, *J* = 10.6 Hz, 1 H), 4.38–4.22 (m, 2 H), 4.1–3.8 (br s, 2 H), 2.10 (s, 3 H).

^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.6, 137.6, 136.1, 135.0, 132.1, 130.7, 129.4, 128.7, 128.6, 128.2, 127.5, 126.6, 120.5, 74.6, 17.5.

HRMS (ESI): *m/z* calc. for $C_{18}H_{20}NO_2$ [M + H]$^+$: 282.1494; found: 282.1519.

***N*-(Allyloxy)-*N*-(2-methoxyphenyl)-2-phenylacetamide (3s2)[36]**
Colorless oil; yield: 23 mg (38%).

IR (film): 3029, 2940, 1685, 1597, 1497, 1368, 1281, 1257, 1023, 756, 726 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.51–7.39 (m, 1 H), 7.38–7.07 (m, 7 H), 7.05–6.95 (m, 1 H), 5.90 (ddt, *J* = 17.2, 10.5, 6.3 Hz, 1 H), 5.32 (d, *J* = 17.2 Hz, 1 H), 5.23 (d, *J* = 10.5 Hz, 1 H), 4.44–4.26 (m, 2 H), 4.1–3.5 (br s, 2 H), 3.75 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 155.7, 134.9, 131.0, 129.3, 128.3, 126.6, 120.7, 120.1, 112.2, 75.3, 55.7, 40.6.

HRMS (ESI): *m/z* [M + H]$^+$ calcd for $C_{18}H_{20}NO_3$: 298.1443; found: 298.1434.

***N*-(Benzyloxy)-*N*-(2-methoxyphenyl)-2-phenylacetamide (3s3)[36]**
Colorless oil; yield: 17 mg (24%).

IR (film): 3030, 2940, 1683, 1496, 1454, 1369, 1281, 1024, 755, 726, 697 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-d_6): δ = 7.45 (t, *J* = 7.8 Hz, 1 H), 7.40–7.05 (m, 12 H), 7.02 (dt, *J* = 7.8, 1.2 Hz, 1 H), 4.87 (s, 2 H), 3.75 (s, 3 H), 4.0–3.4 (br s, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 155.4, 134.7, 130.8, 129.4, 129.1, 128.4, 128.2, 128.1, 126.5, 120.6, 111.9, 76.1, 55.7, 40.6.

HRMS (ESI): *m/z* [M + Na]$^+$ calcd for $C_{22}H_{21}NO_3$ + Na: 370.1419; found: 370.1414.

2-Bromo-*N*-methoxy-*N*-{2-[(methoxyamino)carbonyl]phenyl}benzamide (6)
White solid; mp 145–147 °C; yield: 34 mg (88%).

IR (film): 3212, 2984, 1668, 1662, 1377, 1024, 760 cm^{-1}.

^1H NMR (400 MHz, DMSO-d_6): δ = 11.7–11.5 (br s, 1 H), 7.79–7.32 (m, 7 H), 7.26–7.06 (m, 1 H), 3.68 (s, 3 H), 3.43 (s, 3 H).

^{13}C NMR (100 MHz, DMSO-d_6): δ = 166.1, 163.7, 137.4, 134.8, 132.2, 131.2, 131.1, 130.9, 128.7, 128.4, 127.5, 126.8, 118.4, 62.9, 61.6.

Anal. Calcd for $C_{16}H_{15}BrN_2O_4$: C, 50.68; H, 3.99; N, 7.39. Found: C, 50.85; H, 3.90; N, 7.13.

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