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Amide Synthesis

Nickel-Catalyzed Reductive Addition of Aryl/Benzyl Halides and Pseudohalides to Carbodiimides for the Synthesis of Amides

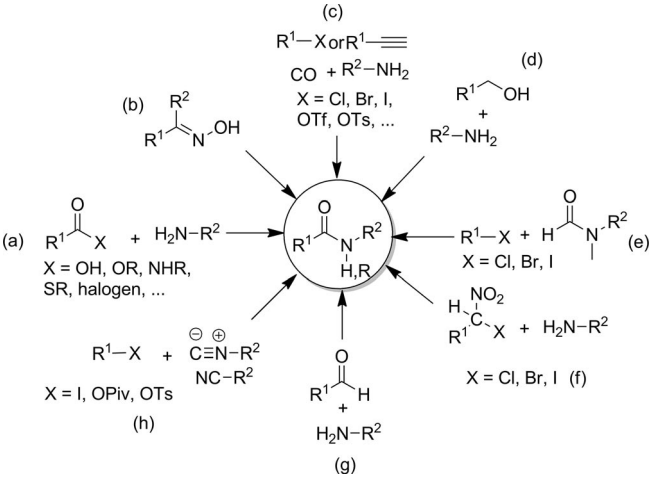
Farhad Panahi,[a] Fereshteh Jamedi,[a] and Nasser Iranpoor*[a]

Abstract: A Nickel-catalyzed reductive process is described for the direct amidation of benzyl and aryl halides using carbodiimides as the amidating agent. Moreover, aryl and benzyl C–O electrophiles such as triflate, acetate, tosylate, trityl ether, and pivalate were converted into amides using this method. The in-situ-generated Ni^0 acts as a catalyst for the reaction at room temperature for benzylic substrates, and 70 °C for aryl electrophiles. This new nickel-catalyzed reductive coupling protocol provides a general and operationally simple method for the synthesis of diverse amides using carbodiimides. Amides bearing bulky substituents can be synthesized by this strategy in high yield, which demonstrates its effectiveness in amide synthesis.

Introduction

The development of new organic transformations to open up simple and efficient pathways for the synthesis of complex molecules is important in organic chemistry.[1] The use of such methods for the synthesis of amides is of great importance, as the amide motif can be used to generate several other organic functionalities.[2] Amides are an important class of organic compounds because of their uses in the synthesis of natural products, advanced materials, and biologically active compounds.[3] Traditional methods for the synthesis of amides are based on the condensation of carboxylic acid derivatives with amines.[4] Despite the simplicity of such methods, they often suffer from several limitations.[5] To avoid the problems associated with these traditional methods, and to enable rwactions with a broad substrate scope for the synthesis of new amides, a great deal of effort has been made towards the development of new protocols. Some of the most widely used strategies in amide synthesis are shown in Scheme 1.[6]

As shown in Scheme 1, most of these approaches used for amide synthesis are based on transition-metal-catalyzed coupling reactions.[6] One interesting method introduced recently by Martin et al. is a reductive amidation approach using a nickel-catalyzed reductive coupling reaction.[7] The reductive coupling strategy is one of the most effective approaches to the formation of carbon–carbon and carbon–heteroatom bonds in modern organic chemistry.[8] Transition-metal-catalyzed reductive cross-coupling reactions facilitate the coupling of two electrophiles by converting of one of them into a nucleophile in situ.[9] This strategy is attractive in terms both of experimental simplicity and of step economy.



Scheme 1. Some synthetic approaches to amides. a) Traditional methods; b) Beckmann rearrangement; c) aminocarbonylation of aryl halides and alkynes; d) acceptorless dehydrogenative coupling reactions; e) cross-coupling of formamides with alkyl/aryl halides; f) umpolung reaction of amines with α-halo nitro alkanes; g) oxidative amidation of aldehydes with amines; h) amidation using isocyanides and nitriles.

As an example of this approach, the direct coupling of aryl/alkyl halides and carbonyl compounds, both electrophilic substrates that are readily available with a wide variety of structures, is possible.[10] In work by Martin et al., isocyanates were used as amidating agents, and reductive coupling of different aryl and benzyl electrophiles with isocyanates resulted in the production of amides. Also, we have previously studied a Ni-catalyzed reductive coupling process for the addition of benzylic substrates to aldehydes for the synthesis of diverse benzylic alcohols.[11] In a continuation of our work on Ni-catalyzed reductive coupling,[11] we found that amides could be produced by the reductive coupling of aryl/benzyl halides and carbodiimides. In this new strategy, carbodiimides were used instead of isocyanates in order to increase the variety of amidating agents

[a] *Department of Chemistry, College of Sciences, Shiraz University, Shiraz 71454, Iran*
E-mail: iranpoor@chem.susc.ac.ir

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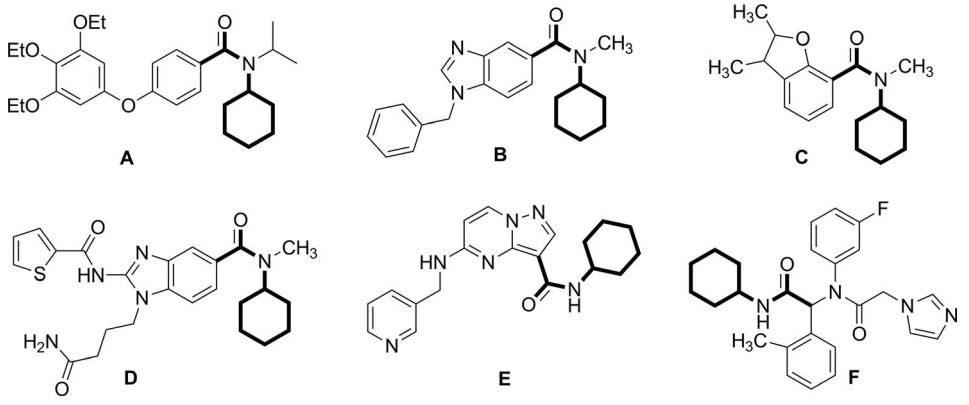
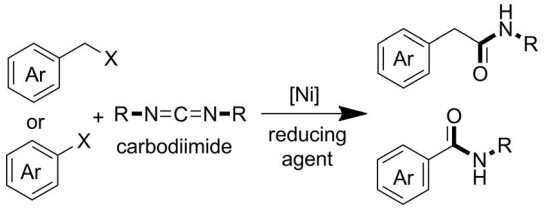


Figure 1. Structures of some biologically active compounds incorporating an *N*-cyclohexyl amide moiety.

that could be used in the reductive amidation process, and also to avoid some of the problems associated with the use of isocyanates in organic transformations.[12] Carbodiimides are an important class of reagents for organic synthesis due to their ready availability and their utility in coupling reactions.[13] Due to the high reactivity of the carbon atom in carbodiimides, it can be used as a counterpart electrophile in reductive coupling reactions. Our new strategy resulted in the generation of amides (Scheme 2), and, to the best of our knowledge, there is no previous report in the literature on the use of carbodiimides for the synthesis of amides through a nickel-catalyzed reductive process.



Scheme 2. Our new strategy for the synthesis of amides using the reductive coupling of aryl or benzyl halides and carbodiimides.

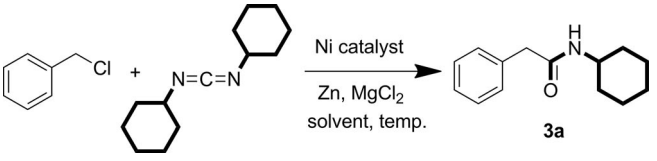
One of the most readily available carbodiimides is DCC (dicyclohexylmethanediimine). The reaction between benzylic substrates and DCC resulted in the production of *N*-cyclohexyl-2-phenylacetamides. This class of compounds has many biological and synthetic applications. The structures of some biologically active compounds containing an *N*-cyclohexyl amide moiety are shown in Figure 1.[14]

In addition, this class of amides is also important in organic synthesis. For example, a range of *N*-cyclohexyl amides have been used by Bechara et al. for the chemoselective synthesis of ketones and ketimines.[15]

Results and Discussion

We began by studying the reaction of benzyl chloride (**1a**) and dicyclohexylmethanediimine (DCC, a cheap and accessible carbodiimide) as model substrates to find the optimum conditions (Table 1).

Table 1. Optimization of reaction conditions for the Ni-catalyzed amidation of benzyl chloride with DCC.[a]



Entry	Ni catalyst (mol-%)	Solvent	*T* [°C]	Time [h]	Yield [%][b]
1	–	DMF	r.t.	24	0
2	$NiCl_2{\cdot}5H_2O$ (10 mol-%)	DMF	r.t.	24	55
3	$NiCl_2(PPh_3)_2$ (10 mol-%)	DMF	r.t.	24	61
4	$NiCl_2(PCy_3)_2$ (10 mol-%)	DMF	r.t.	24	76
5	$NiCl_2$(dppe) (10 mol-%)	DMF	r.t.	24	75
6	$NiCl_2$(dppp) (10 mol-%)	DMF	r.t.	24	80
7	$NiCl_2$(dppf) (10 mol-%)	DMF	r.t.	24	85
8	$NiCl_2$(dppf) (10 mol-%)	DMF	50	24	86
9	$NiCl_2$(dppf) (10 mol-%)	DMSO	r.t.	24	50
10	$NiCl_2$(dppf) (10 mol-%)	THF	r.t.	24	58
11	$NiCl_2$(dppf) (10 mol-%)	toluene	r.t.	24	40
12	$NiCl_2$(dppf) (5 mol-%)	DMF	r.t.	24 (48)	78 (79)
13	$NiCl_2$(dppf) (7.5 mol-%)	DMF	r.t.	24 (30)	82 (84)
14	$NiCl_2$(dppf) (12 mol-%)	DMF	r.t.	24	85
15	$NiCl_2$(dppf) (10 mol-%)	DMF	r.t.	24	84[c]
16	$NiCl_2$(dppf) (10 mol-%)	DMF	r.t.	24	75[d]
17	$NiCl_2$(dppf) (10 mol-%)	DMF	r.t.	24	0[e]
18	$NiCl_2$(dppf) (10 mol-%)	DMF	r.t.	24	43[f]

[a] Reaction conditions: benzylic substrate (1.0 mmol), DCC (1.2 mmol), Zn (3.0 mmol), $MgCl_2$ (2.0 mmol), solvent (3.0 mL); dppe = 1,2-bis(diphenylphosphino)ethane; dppp = 1,3-bis(diphenylphosphino)propane; dppf = 1,1′-bis(diphenylphosphino)ferrocene. [b] Isolated yield. [c] 2.5 mmol Zn was used. [d] Mn was used instead of Zn. [e] Zn was not used. [f] $MgCl_2$ was not used.

In the absence of Ni catalyst, no product was observed (Table 1, entry 1). However, in the presence of $NiCl_2$ as catalyst, a 55 % yield of the product was obtained (Table 1, entry 2). To try to improve the reaction yield, other Ni catalysts bearing phosphine ligands were tested. When $NiCl_2$(dppf) was used, an 85 % yield of the product was obtained (Table 1, entries 3–7). When the temperature was increased to 50 °C, no improvement in the reaction yield was observed, thus room temperature was selected as optimum (Table 1, entry 8). Different solvents were tested, but no better solvent was found, and DMF selected as

the best solvent tested (Table 1, entries 9–11). Different catalyst loadings were tested, and a catalyst loading of 10 mol-% of $NiCl_2$(dppf) was found to be optimal (Table 1, entries 12–14). Increasing the amount of Zn did not have a significant effect on the reaction yield (Table 1, entry 15). When Mn was used instead of Zn, no improvement in the reaction yield was observed (Table 1, entry 15). In the absence of the Zn reducing agent, no product was observed. According to the literature, the role of Zn in this reaction is to reduce Ni^{II} to Ni^{I}/Ni^{0}.[16] When the $MgCl_2$ was removed, the yield of the reaction decreased to 43 %, so this additive has a significant effect on the reaction yield. It has been reported previously that in this type of reaction, the use of $MgCl_2$ as an additive improved the reaction yields.[11,17]

The results of optimization process (Table 1) show that the maximum yield of product **3a** was obtained in the presence of $NiCl_2$(dppf) (10 mol-%) as catalyst, Zn (3 mmol) as reducing agent, and $MgCl_2$ (2 mmol) as additive, in DMF (as solvent), at room temperature.

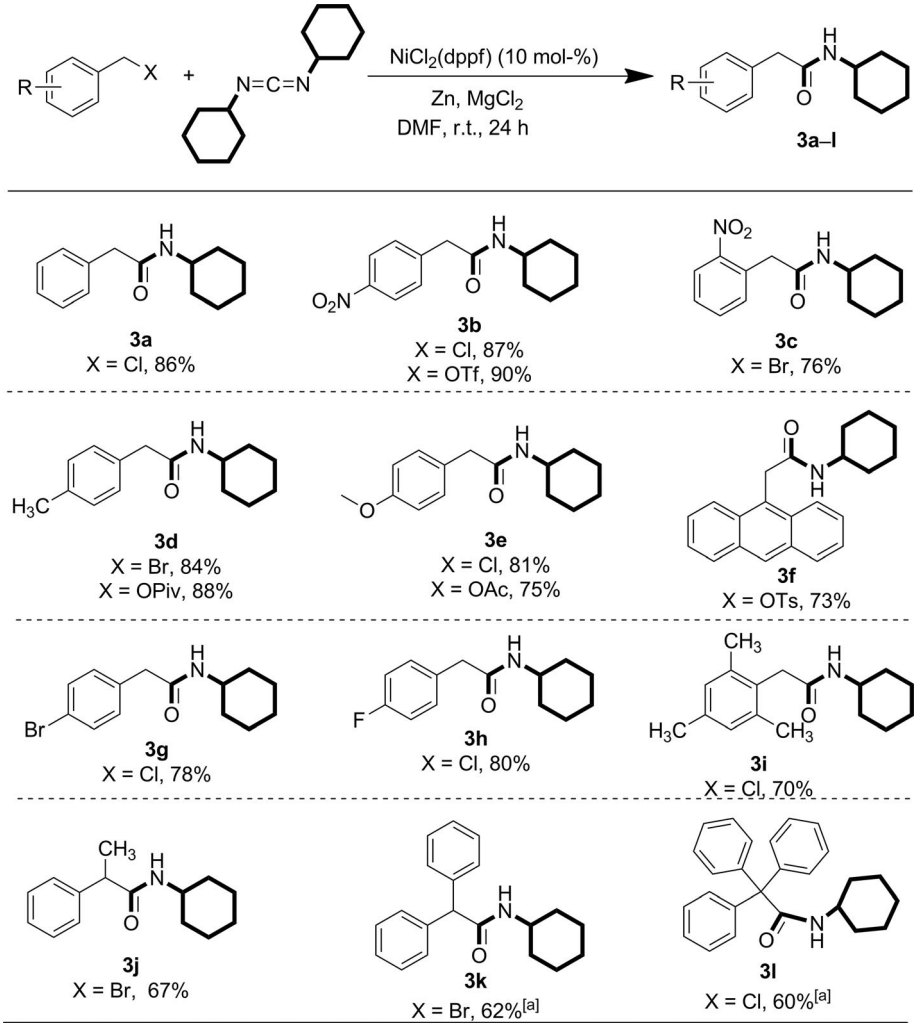
There is a great variety of benzylic C–O electrophiles, and they can be used as readily available alternatives to benzylic halides. Next, we examined the generality of the reaction using other benzylic substrates under the optimized reaction conditions, as shown in Table 2.

Table 2. Different benzylic substrates.[a]

Entry	X	Time [h]	Yield [%][b]
1	Cl	24	86
2	Br	20	87
3	I	18	90
4	OTf	24	85
5	OAc	24	76
6	OTs	24	78
7	OTr	24	65
8	OPiv	24	85
9	OH	24	0

[a] Reaction conditions: benzylic substrate (1.0 mmol), DCC (1.2 mmol), Zn (3.0 mmol), $MgCl_2$ (2.0 mmol), $NiCl_2$(dppf) (10.0 mol-%), DMF (3.0 mL). [b] Isolated yield.

We found that, in addition to benzyl halides (Cl, Br, and I), other benzylic C–O electrophiles such as triflate, acetate, tosylate, trityl ether, and pivalate also resulted in the formation of the desired product. This point reveals the importance of our



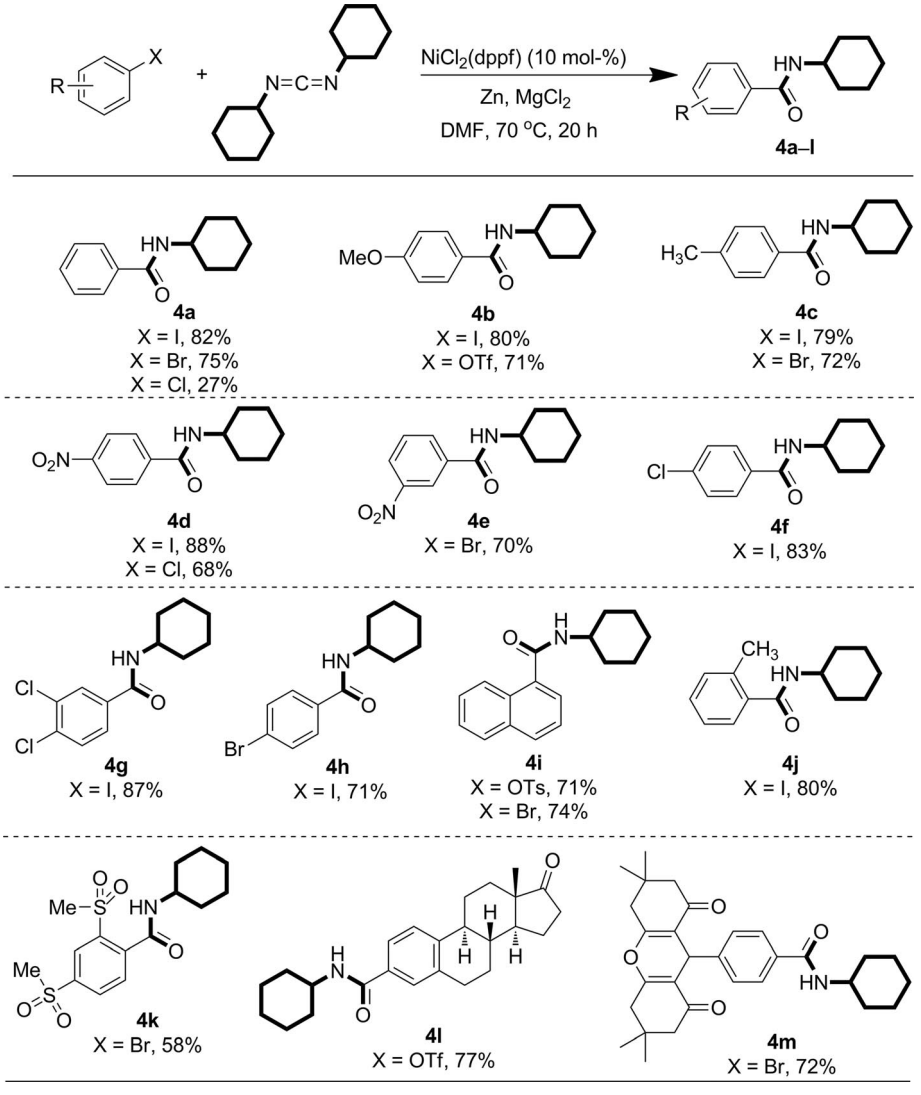
Scheme 3. Ni-catalyzed reductive amidation of benzylic substrates with DCC. Reaction conditions: benzylic substrate (1.0 mmol), DCC (1.2 mmol), Zn (3.0 mmol), $MgCl_2$ (2.0 mmol), $NiCl_2$(dppf) (10.0 mol-%), DMF (3.0 mL); all yields refer to isolated products. [a] The reaction was carried out at 50 °C.

protocol for the synthesis of diverse amides using a range of benzylic substrates. All of the used benzylic C–O electrophiles gave the amide product in good yields. Benzyl alcohol failed to produce the desired amide and remained unchanged.

To show the generality of this protocol in the synthesis of other *N*-cyclohexyl amides, other benzylic derivatives were also tested, and the results are shown in Scheme 3. Different types of benzylic substrates can be used in this approach. Benzylic substrates bearing electron-rich and electron-poor substituents were both tolerated, and excellent yields of the desired products (i.e., **3b**–**3e**) were obtained. When an anthracene derivative was used, the reaction occurred readily to give amide **3f** in good yield. To demonstrate the applicability of this reaction with halogen-substituted substrates, 4-bromobenzyl chloride and 4-fluorobenzyl chloride were also examined, and good yields of the products (i.e., **3g** and **3h**) were obtained. 2,4,6-Trimethylbenzyl chloride was also used as a sterically hindered substrate, and compound **3i** was obtained in 70 % isolated yield. We also examined secondary and tertiary benzylic substrates under the optimized conditions. The use of (1-bromoethyl)benzene, (bromomethylene)dibenzene, and trityl chloride resulted in the formation of the corresponding products (i.e., **3j**–**3l**) in 67, 62, and 60 % yields, respectively.

The method was also used for the introduction of the amide group into aromatic systems, bearing in mind the importance of benzamides as valuable building blocks in medicinal chemistry.[18] In fact, synthetic routes often require the conversion of aryl halides to amides, and this encouraged us to explore the possibility of using our method for this conversion.[19] Thus, iodobenzene and DCC were subjected to the optimized conditions at room temperature, but no product was observed. Fortunately, at the higher temperature of 70 °C, *N*-cyclohexylbenzamide was produced in 82 % yield. Different conditions were also tested, but no improvement was observed. Thus, the optimized conditions could also be used for the amidation of aryl substrates using DCC.

To establish the scope of this new method, a number of aromatic substrates were tested under the optimized conditions



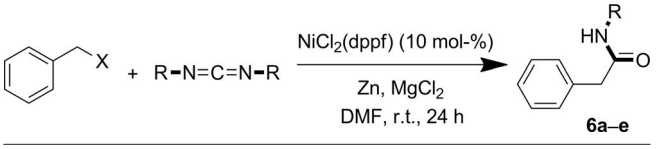
Scheme 4. Ni-catalyzed reductive amidation of aryl halides with DCC. Reaction conditions: aryl–X (1.0 mmol), DCC (1.2 mmol), Zn (3.0 mmol), $MgCl_2$ (2.0 mmol), $NiCl_2$(dppf) (10.0 mol-%), DMF (3.0 mL). All yields refer to isolated products.

(Scheme 4). A range of substrates bearing an electron-donating (**4b** and **4c**) or an electron-withdrawing (**4d** and **4e**) group on the phenyl ring gave the corresponding amides in good yields. Some substrates bearing two halogen groups were also tested, and these gave the desired monoamides (i.e., **4f**–**4h**) in relatively good yields, with selectivity between the two different halogens. 1-Bromonaphthalene was found to react, and the corresponding amide (i.e., **4i**) was obtained in 74 % yield. Naphthalen-1-tosylate also gave **4i** in 71 % yield. 2-Iodotoluene was produced *N*-cyclohexyl-2-methylbenzamide (**4j**) in 80 % yield. 1-Bromo-2,4-bis(methylsulfonyl)benzene, bearing an SO_2Me group, was also compatible with the optimized conditions and gave **4k**. A triflate derivative of estrone reacted with DCC under the optimized conditions to give the corresponding amide derivative (i.e., **4l**) in 77 % yield.[20] Also, 9-(4-bromophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-dione was used in this reaction, and amide **4m** was produced in 72 % yield.[21]

Encouraged by these results, we examined the scope of our Ni-catalyzed reductive amidation with other carbodiimides to investigate the generality of this approach to amide synthesis (Scheme 5). We found that the reaction of aryl halides with bis(trimethylsilyl)methanediimine proceeded smoothly to give primary amides **5a**–**5c** in good yields. When di-*p*-tolylmethanediimine was used, the corresponding amides (i.e., **5d**–**5f**) were obtained in yields of 71, 73, and 70 %, respectively. Treatment of aryl halides and aryl C–O electrophiles with *tert*-butyl, isopropyl, and ethyl carbodiimides gave the corresponding amide products in good yields. These results demonstrate that this protocol can be used with different carbodiimides, thus representing an efficient route for the synthesis of diverse substituted amides.

The scope of the reaction was further investigated by using benzylic substrates instead of aryl halides (Table 3). We found that the protocol was efficient for synthesis of structurally different amides using benzylic substrates with carbodiimides. For carbodiimides with aromatic and bulky aliphatic substituents, such as *tert*-butyl, the yields were lower than with other substrates.

Table 3. Amidation of benzylic substrates with different carbodiimides.[a]

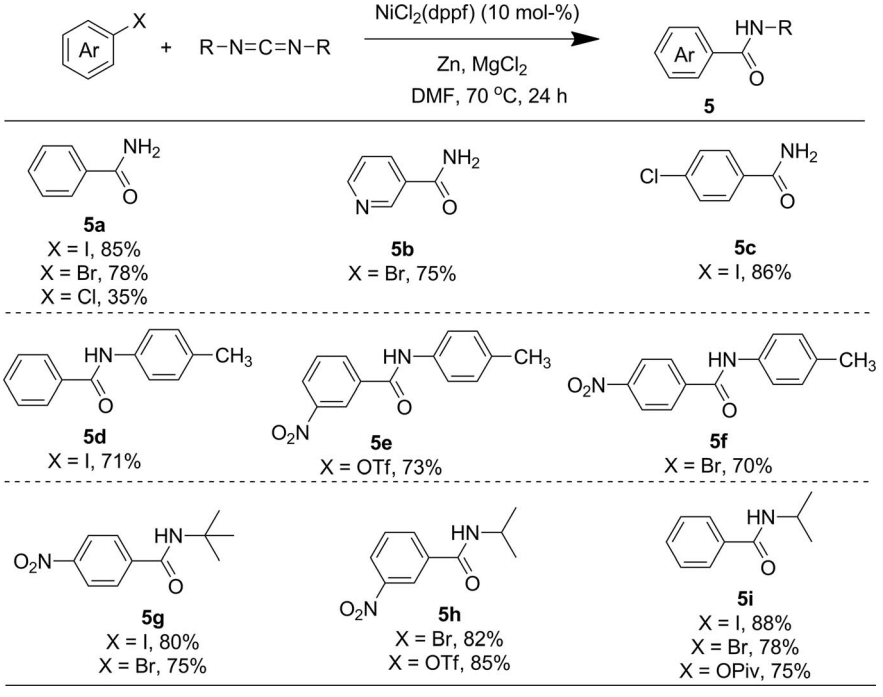


6a–e

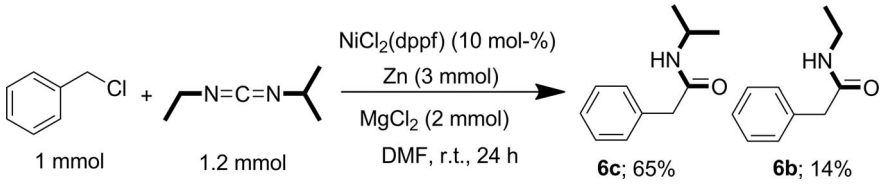
Entry	X	R	Product **6**	Yield [%][b]
1	Cl	H	**6a**	90
2	OTs	H	**6a**	85
3	OPiv	H	**6a**	88
4	Cl	Et	**6b**	91
5	OTf	Et	**6b**	89
6	Br	*i*Pr	**6c**	90
7	OTf	*i*Pr	**6c**	87
8	Cl	*t*Bu	**6d**	84
9	OTf	*t*Bu	**6d**	82
10	Cl	*p*-tolyl	**6e**	72
11	OPiv	*p*-tolyl	**6e**	70

[a] Reaction conditions: benzylic substrate (1 mmol), DCC (1.2 mmol), Zn (3 mmol), $MgCl_2$ (2 mmol), $NiCl_2$(dppf) (10 mol-%), DMF (3 mL). [b] Isolated yield.

An unsymmetrical carbodiimide was also tested in this procedure, and cleavage of the C=N bond connected to the less bulky substituent was observed (Scheme 6).[22]

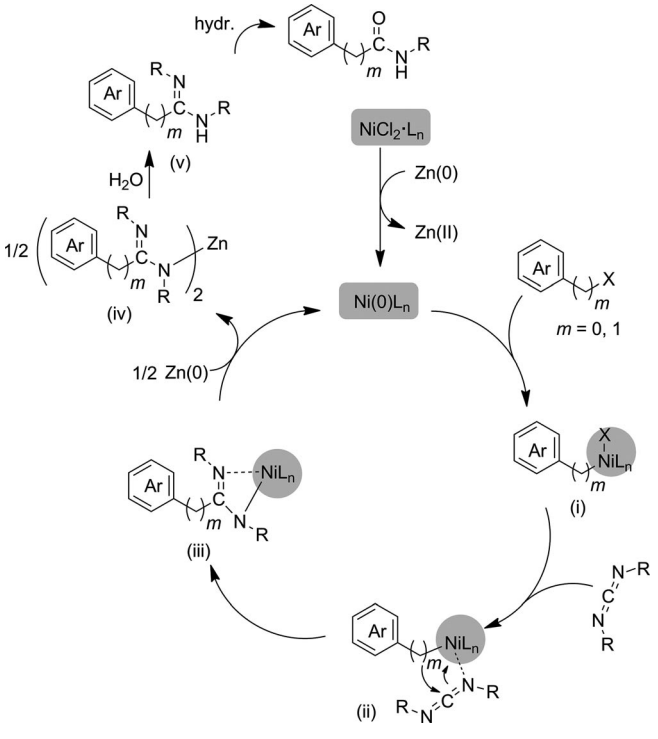


Scheme 5. Amidation of aryl substrates with different carbodiimides. Reaction conditions: aryl substrate (1.0 mmol), DCC (1.2 mmol), Zn (3.0 mmol), $MgCl_2$ (2.0 mmol), $NiCl_2$(dppf) (10.0 mol-%), DMF (3.0 mL). All yields refer to isolated products.



Scheme 6. Use of an unsymmetrical carbodiimide.

Although a detailed mechanistic understanding of this reaction requires further studies, a plausible reaction mechanism for benzylic substrates based on the literature is proposed (Scheme 7).[11,16b] The catalytic cycle is initiated by the reduction of Ni^{II} to Ni^0. It seems that in the presence of Zn, Ni^{II} is reduced to Ni^0, because in the absence of Zn the reaction did not take place.[11] Furthermore, the reaction did take place when $Ni(PPh_3)_2(CO)_2$ was used as an Ni^0 source. These results confirm that this is a Ni^0-catalyzed process. The Ni^0 species undergoes oxidative addition with the C–X bond to form Ni^{II} complex **i**. Then, the carbodiimide coordinates with complex **i** to form adduct **ii**. Next, the aryl or benzyl moiety adds to the activated carbodiimide to form intermediate **iii**.[16a] Zn can then regenerate the Ni^0 complex to continue the catalytic cycle. Protonation of adduct **iv** results in the formation of amidine product **v**.[23] The amidine product was isolated from the reaction mixture. Further hydrolysis of the amidine resulted in the formation of the amide.[22] It should be mentioned that for benzylic substrates the formation of Ni^I complexes in the presence of Zn has been reported; this represents another possible pathway for the formation of intermediate **iii** from **i**.[24]



Scheme 7. Proposed reaction mechanism for Ni-catalyzed amide synthesis using carbodiimides.

Conclusions

In conclusion, we have established the first examples of the Ni-catalyzed addition of benzylic and aryl substrates to carbodiimides using a Ni/Zn catalytic system for synthesis of amide derivatives. The reaction proceeds with a catalytic amount of Ni under mild conditions, and the procedure was also applicable to C–O electrophiles. This method is characterized by its operational simplicity, wide preparative scope, and the ready availability of the substrates used. The method also represents a simple choice for the synthesis of amides using carbodiimides as an easily handled amidating agent without the need for the preparation of sensitive organometallic reagents.

Experimental Section

General Remarks: Chemicals were purchased from Fluka, Merck, and Aldrich, and were used without further purification. Known products were characterized by comparison of their spectral and physical data with those reported in the literature. ^1H (250 MHz) and ^{13}C (62.9 MHz) NMR spectra were recorded with a Bruker Avance spectrometer in $CDCl_3$ and [D_6]DMSO solutions, and tetramethylsilane (TMS) was used as an internal standard. FTIR spectra were recorded with a Shimadzu FTIR 8300 spectrophotometer. Melting points were determined in open capillary tubes with a Barnstead electro-thermal 9100 BZ circulating oil melting-point apparatus. Reactions were monitored by TLC on silica gel PolyGram SILG/UV254 plates. Column chromatography was carried out on columns of silica gel 60 (70–230 mesh).

General Procedure for Ni-Catalyzed Reductive Amidation: A mixture of carbodiimide (1.2 mmol), aryl/benzyl substrate (1.0 mmol), Zn (3.0 mmol), $MgCl_2$ (2.0 mmol), and $NiCl_2$(dppf) (10 mol-%) was stirred in dry DMF (3.0 mL) in a 50 mL flask for 24 h at room temp. or 70 °C. After TLC indicated that the reaction was complete, HCl (0.5 M aq; 25 mL) was added, and the mixture was stirred for 3 h. Then ethyl acetate (50 mL) and saturated $NaHCO_3$ (25 mL) were added to the mixture. The aqueous and organic layers were separated, then the aqueous phase was extracted with ethyl acetate (2 × 25 mL). The combined organic layers were then dried with anhydrous Na_2SO_4, filtered, and concentrated under vacuum. The crude product was purified by column chromatography (*n*-hexane/ethyl acetate) to give the desired pure product.

***N*-Cyclohexyl-2-phenylacetamide (3a):**[25] Yield 86 % (186 mg). ^1H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.70–1.89 (m, 10 H), 3.39–3.43 (m, 1 H), 3.98–3.99 (m, 2 H), 6.67 (s, 1 H), 7.30–7.39 (m, 3 H), 7.55–7.63 (m, 2 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.9, 25.6, 32.4, 44.2, 127.6, 129.0, 129.6, 135.2, 170.0 ppm. $C_{14}H_{19}NO$ (217.31): calcd. C 77.38, H 8.81, N 6.45; found C 77.31, H 8.76, N 6.37.

***N*-Cyclohexyl-2-(4-nitrophenyl)acetamide (3b):** Yield 90 % (236 mg). ^1H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.1–2-1.93 (m, 10 H), 3.43–3.48 (m, 1 H), 4.12–4.15 (m, 2 H), 7.25–7.28 (m, 3 H), 8.11–8.14 (m, 2 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.9, 25.6, 33.9,

43.1, 49.1, 124.0, 129.1, 141.0, 146.0, 171.6 ppm. $C_{14}H_{18}N_2O_3$ (262.31): calcd. C 64.11, H 6.92, N 10.68; found C 63.97, H 6.83, N 10.76.

***N*-Cyclohexyl-2-(2-nitrophenyl)acetamide (3c):** Yield 76 % (199 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.07–1.89 (m, 10 H), 3.38–3.42 (m, 1 H), 4.02–4.05 (m, 2 H), 7.55–7.57 (m, 2 H), 7.71–7.72 (m, 1 H), 8.05 (s, 1 H), 9.91 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.9, 25.6, 33.9, 38.6, 49.1, 125.6, 128.5, 128.9, 130.6, 135.3, 149.8, 171.3 ppm. $C_{14}H_{18}N_2O_3$ (262.31): calcd. C 64.11, H 6.92, N 10.68; found C 64.01, H 6.84, N 10.59.

***N*-Cyclohexyl-2-(*p*-tolyl)acetamide (3d):** Yield 88 % (203 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.99–1.87 (m, 10 H), 2.10 (s, 3 H), 3.41 (s, 1 H), 3.91 (s, 2 H), 7.04–7.06 (m, 2 H), 7.40–7.43 (m, 2 H), 7.72 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 21.3, 24.9, 25.6, 32.3, 43.5, 50.0, 229.5, 129.60, 129.61, 129.65, 132.6, 137.3, 171.4 ppm. $C_{15}H_{21}NO$ (231.34): calcd. C 77.88, H 9.15, N 6.05; found C 77.79, H 9.09, N 6.00.

***N*-Cyclohexyl-2-(4-methoxyphenyl)acetamide (3e):** Yield 81 % (200 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.09–1.88 (m, 10 H), 3.39–3.84 (m, 1 H), 3.84 (s, 3 H), 4.01 (d, J = 1.5 Hz, 2 H), 6.90 (d, J = 6.0 Hz, 2 H), 6.90 (d, J = 6.2 Hz, 2 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.9, 25.7, 32.3, 43.7, 49.9, 55.8, 115.0, 127.9, 130.7, 159.8, 171.5 ppm. $C_{15}H_{21}NO_2$ (247.34): calcd. C 72.84, H 8.56, N 5.66; found C 72.76, H 8.51, N 5.59.

2-(Anthracen-9-yl)-*N*-cyclohexylacetamide (3f): Yield 73 % (231 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.00–1.98 (m, 10 H), 3.39–3.43 (m, 1 H), 4.93 (s, 2 H), 7.49–7.54 (m, 4 H), 7.86–7.89 (m, 3 H), 8.30–8.32 (m, 2 H), 8.74 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.7, 25.3, 32.3, 37.5, 50.1, 124.9, 125.0, 127.2, 127.9, 128.1, 128.9, 129.1, 170.3 ppm. $C_{22}H_{23}NO$ (317.43): calcd. C 83.24, H 7.30, N 4.41; found C 83.16, H 7.22, N 4.33.

2-(4-Bromophenyl)-*N*-cyclohexylacetamide (3g): Yield 78 % (231 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.87–1.88 (m, 10 H), 3.38–3.42 (m, 1 H), 3.98 (d, J = 4.2 Hz, 2 H), 6.88–6.94 (m, 2 H), 7.27–7.33 (m, 3 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.9, 25.6, 29.6, 33.9, 37.0, 49.1, 120.4, 130.2, 131.4, 140.1, 182.9 ppm. $C_{14}H_{18}BrNO$ (296.21): calcd. C 56.77, H 6.13, N 4.73; found C 56.68, H 6.05, N 4.65.

***N*-Cyclohexyl-2-(4-fluorophenyl)acetamide (3h):** Yield 80 % (188 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.78–1.89 (m, 10 H), 3.65–3.67 (m, 1 H), 4.04 (t, J = 2.0 Hz, 2 H), 6.75–7.05 (m, 4 H), 7.35 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.8, 25.5, 33.7, 45.1, 51.5, 115.1, 115.4, 127.5, 127.6, 130.9, 131.0, 167.9, 174.4 ppm. $C_{14}H_{18}FNO$ (235.30): calcd. C 71.46, H 7.71, N 5.95; found C 71.34, H 7.63, N 5.85.

***N*-Cyclohexyl-2-mesitylacetamide (3i):** Yield 70 % (181 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.09–1.87 (m, 10 H), 2.81 (s, 3 H), 2.98 (s, 6 H), 3.32–3.35 (m, 1 H), 3.77 (s, 2 H), 7.38 (s, 1 H), 7.95 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 16.5, 21.8, 24.8, 25.5, 32.1, 35.5, 51.0, 127.6, 133.0, 137.0, 171.8 ppm. $C_{17}H_{25}NO$ (259.39): calcd. C 78.72, H 9.71, N 5.40; found C 78.63, H 9.64, N 5.31.

***N*-Cyclohexyl-2-phenylpropanamide (3j):** Yield 67 % (154 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.85–1.69 (m, 13 H), 4.11–4.14 (m, 1 H), 4.26 (s, 1 H), 7.52–7.80 (m, 5 H), 8.20 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 15.4, 24.7, 25.1, 31.2, 43.4, 50.8, 126.9, 128.6, 136.9, 171.4 ppm. $C_{15}H_{21}NO$ (231.34): calcd. C 77.88, H 9.15, N 6.05; found C 77.76, H 9.06, N 5.94.

***N*-Cyclohexyl-2,2-diphenylacetamide (3k):** Yield 67 % (154 mg). CAS No: 24932–56–7. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.08–2.10 (m, 10 H), 4.00 (s, 1 H), 5.07 (s, 1 H), 7.31–7.39 (m, 4 H), 7.67–7.72 (m, 6 H), 9.81 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.7, 25.1, 31.9, 50.8, 58.3, 126.9, 128.6, 129.3, 138.7, 171.4 ppm. $C_{20}H_{23}NO$ (293.41): calcd. C 81.87, H 7.90, N 4.77; found C 81.76, H 7.81, N 4.69.

***N*-Cyclohexyl-2,2,2-triphenylacetamide (3l):** Yield 60 % (221 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.86–1.94 (m, 10 H), 3.46–3.48 (m, 1 H), 7.19–7.54 (m, 15 H), 8.05 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.9, 25.6, 33.9, 49.1, 62.0, 126.2, 129.3, 129.4, 143.2, 174.6 ppm. $C_{26}H_{27}NO$ (369.51): calcd. C 84.51, H 7.37, N 3.79; found C 84.42, H 7.31, N 3.70.

***N*-Cyclohexyl Benzamide (4a):**[26] Yield 82 % (166 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.96–1.70 (m, 10 H), 4.03–4.06 (m, 1 H), 7.23–7.28 (m, 2 H), 7.54–7.64 (m, 2 H), 9.59 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.4, 24.5, 33.1, 54.6, 126.6, 129.8, 131.9, 134.5, 166.6 ppm. $C_{13}H_{17}NO$ (203.29): calcd. C 76.81, H 8.43, N 6.89; found C 76.72, H 8.37, N 6.81.

***N*-Cyclohexyl-4-methoxybenzamide (4b):**[15] Yield 80 % (186 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.02–1.97 (m, 10 H), 3.86 (s, 3 H), 4.03–4.09 (m, 1 H), 7.02–7.07 (m, 2 H), 7.20–7.26 (m, 2 H), 9.44 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.4, 24.7, 33.3, 52.7, 54.8, 115.5, 117.6, 128.6, 129.9, 162.5, 165.4 ppm. $C_{14}H_{19}NO_2$ (233.31): calcd. C 72.07, H 8.21, N 6.00; found C 72.01, H 8.16, N 5.91.

***N*-Cyclohexyl-4-methylbenzamide (4c):** Yield 79 % (171 mg). CAS No: 53205–68–8. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.90–1.88 (m, 10 H), 2.42 (s, 3 H), 3.96 (s, 1 H), 7.11–7.13 (m, 2 H), 7.32 (d, J = 7.5 Hz, 2 H), 9.85 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 17.4, 29.3, 30.4, 49.2, 128.2, 128.4, 131.7, 131.8, 174.8 ppm. $C_{14}H_{19}NO$ (217.31): calcd. C 77.38, H 8.81, N 6.45; found C 77.31, H 8.75, N 6.37.

***N*-Cyclohexyl-4-nitrobenzamide (4d):**[15] Yield 88 % (218 mg). CAS No. 7506–46–9. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.05–1.96 (m, 10 H), 3.50–3.52 (m, 1 H), 7.96–7.99 (m, 2 H), 8.34–8.37 (m, 2 H), 8.91 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.6, 24.9, 32.6, 52.6, 118.9, 123.9, 132.7, 153.6, 175.1 ppm. $C_{13}H_{16}N_2O_3$ (248.28): calcd. C 62.89, H 6.50, N 11.28; found C 62.80, H 6.43, N 11.22.

***N*-Cyclohexyl-3-nitrobenzamide (4e):** Yield 70 % (173 mg). CAS No. 2702–32–1. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.90–1.59 (m, 10 H), 3.54 (br. s, 1 H), 7.48–7.53 (m, 1 H), 8.05–8.07 (m, 2 H), 8.37–8.44 (m, 2 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.8, 25.1, 32.2, 48.6, 121.9, 125.4, 129.7, 133.7, 136.1, 147.5, 163.0 ppm. $C_{13}H_{16}N_2O_3$ (248.28): calcd. C 62.89, H 6.50, N 11.28; found C 62.80, H 6.41, N 11.22.

4-Chloro-*N*-cyclohexylbenzamide (4f): Yield 73 % (173 mg). CAS No. 57707–20–7. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.09–1.77 (m, 10 H), 3.72 (br. s, 1 H), 7.46 (d, J = 5.5 Hz, 2 H), 7.83–7.86 (m, 2 H), 8.25 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.8, 25.2, 32.3, 48.4, 128.0, 129.1, 133.5, 135.7, 164.1 ppm. $C_{13}H_{16}ClNO$ (237.73): calcd. C 65.68, H 6.78, N 5.89; found C 65.60, H 6.71, N 5.82.

3,4-Dichloro-*N*-cyclohexylbenzamide (4g): Yield 87 % (236 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.93–1.94 (m, 10 H), 3.40–3.44 (m, 1 H), 7.64–7.88 (m, 3 H), 8.21 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.4, 24.5, 33.1, 54.5, 128.1, 129.0, 130.4, 133.1, 133.5, 136.4, 167.8 ppm. $C_{13}H_{15}Cl_2NO$ (272.17): calcd. C 57.37, H 5.56, N 5.15; found C 57.28, H 5.51, N 5.06.

4-Bromo-*N*-cyclohexylbenzamide (4h):[15] Yield 71 % (200 mg). CAS number: 223553-87-5. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.92–

1.65 (m, 10 H), 4.01–4.07 (m, 1 H), 7.15 (d, *J* = 8.5 Hz, 2 H), 7.70 (d, *J* = 8.2 Hz, 2 H), 8.62 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.4, 24.5, 33.1, 54.7, 126.6, 128.2, 131.9, 133.3, 176.2 ppm. $C_{13}H_{16}BrNO$ (282.18): calcd. C 55.33, H 5.72, N 4.96; found C 55.33, H 5.72, N 4.96.

***N*-Cyclohexyl-1-naphthamide (4i):** Yield 74 % (187 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.78–1.61 (m, 10 H), 4.04–4.07 (m, 1 H), 7.36–7.39 (m, 1 H), 7.55–7.65 (m, 3 H), 7.72–7.76 (m, 1 H), 7.93–8.07 (m, 2 H), 10.13 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.4, 32.8, 33.4, 54.7, 123.1, 123.9, 125.1, 125.3, 127.8, 128.7, 129.1, 132.0, 163.1, 181.8 ppm. $C_{17}H_{19}NO$ (253.35): calcd. C 80.60, H 7.56, N 5.53; found C 80.51, H 7.51, N 5.42.

***N*-Cyclohexyl-2-methylbenzamide (4j):** Yield 80 % (173 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.98–1.66 (m, 10 H), 2.27 (s, 3 H), 3.38–3.44 (m, 1 H), 7.09–7.49 (m, 3 H), 7.91 (s, 1 H), 9.66 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 21.6, 24.47, 24.53, 29.7, 33.1, 54.5, 126.5, 128.4, 130.4, 132.5, 136.4, 140.3, 168.5 ppm. $C_{14}H_{19}NO$ (217.31): calcd. C 77.38, H 8.81, N 6.45; found C 77.38, H 8.81, N 6.45.

***N*-Cyclohexyl-2,4-bis(methylsulfonyl)benzamide (4k):** Yield 58 % (208 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.88–1.89 (m, 10 H), 3.07 (s, 6 H), 4.09–4.12 (m, 1 H), 7.20 (s, 1 H), 7.79 (d, *J* = 8.0 Hz, 1 H), 8.17–8.20 (d, *J* = 7.7 Hz, 1 H), 8.47 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.9, 25.6, 33.9, 44.3, 49.1, 126.7, 130.8, 132.4, 140.1, 141.1, 142.3, 168.2 ppm. $C_{15}H_{21}NO_5S_2$ (359.46): calcd. C 50.12, H 5.89, N 3.90, S 17.84; found C 50.04, H 5.82, N 3.81, S 17.78.

(8*R*,9*S*,13*S*,14*S*)-*N*-Cyclohexyl-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthrene-3-carboxamide (4l): Yield 77 % (292 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.80 (s, 1 H), 0.88–2.73 (m, 23 H), 3.34 (s, 2 H), 3.72 (s, 1 H), 7.24 (d, *J* = 4.7 Hz, 1 H), 7.68 (d, *J* = 2.7 Hz, 1 H), 7.81 (s, 1 H), 8.82 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 18.7, 24.6, 25.6, 26.3, 30.7, 31.3, 32.9, 34.2, 36.5, 40.5, 48.6, 51.7, 52.5, 54.8, 124.2, 126.9, 131.2, 135.1, 142.3, 168.0, 219.7 ppm. $C_{25}H_{33}NO_2$ (379.54): calcd. C 79.11, H 8.76, N 3.69; found C 79.04, H 8.70, N 3.61.

***N*-Cyclohexyl-4-(3,3,6,6-tetramethyl-1,8-dioxo-2,3,4,5,6,7,8,9-octahydro-1*H*-xanthen-9-yl)benzamide (4m):** Yield 72 % (342 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 0.97 (s, 6 H), 1.06 (s, 6 H), 1.08–1.73 (m, 10 H), 2.12–2.26 (m, 4 H), 2.49 (s, 4 H), 4.08 (s, 1 H), 4.69 (s, 1 H), 7.12–7.19 (m, 2 H), 7.26–7.35 (m, 2 H), 8.82 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 24.9, 25.9, 27.3, 29.3, 31.5, 32.2, 40.8, 50.7, 51.2, 115.1, 127.3, 127.7, 131.1, 147.4, 154.7, 167.8, 196.3 ppm. $C_{30}H_{37}NO_4$ (475.63): calcd. C 75.76, H 7.84, N 2.94; found C 75.68, H 7.76, N 2.85.

Benzamide (5a):[27] Yield 85 % (102 mg). CAS No. 55–21–0. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 7.38–7.48 (m, 3 H), 7.87–7.91 (m, 2 H), 8.02 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 127.4, 128.1, 131.1, 134.1, 168.1 ppm. C_7H_7NO (121.14): calcd. C 69.41, H 5.82, N 11.56; found C 69.34, H 5.78, N 11.51.

Nicotinamide (5b): Yield 75 % (91.5 mg). CAS No. 98–92–0. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 7.44–7.59 (m, 2 H), 7.59 (s, 1 H), 8.15–8.20 (m, 3 H), 8.66–8.67 (m, 1 H), 9.00 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 123.3, 129.6, 135.1, 148.6, 151.8, 166.4 ppm. $C_6H_6N_2O$ (122.13): calcd. C 59.01, H 4.95, N 22.94; found C 58.95, H 4.91, N 22.88.

4-Chlorobenzamide (5c):[27] Yield 86 % (134 mg). CAS No. 619–56–7. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 7.19–7.59 (m, 3 H), 7.84–7.88 (m. 2 H), 8.11 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 128.1, 129.3, 131.0, 136.1, 166.9 ppm. C_7H_6ClNO (155.58): calcd. C 54.04, H 3.89, N 9.00; found C 53.98, H 3.83, N 8.94.

4-Chloro-*N*-(*p*-tolyl)benzamide (5d): Yield 71 % (174 mg). CAS No. 2447–95–2. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 2.25 (s, 3 H), 7.11–7.14 (m, 2 H), 7.46–7.67 (m, 4 H), 7.91–7.95 (m, 2 H), 10.15 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 127.38, 127.42, 128.07, 128.14, 131.1, 134.05, 134.15, 168.1 ppm. $C_{14}H_{12}ClNO$ (245.71): calcd. C 68.44, H 4.92, N 5.70; found C 68.38, H 4.86, N 5.63.

3-Nitro-*N*-(*p*-tolyl)benzamide (5e): Yield 73 % (187 mg). CAS No: 6911–92–8. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 2.27 (s, 3 H), 7.17 (d, *J* = 8.2 Hz, 2 H), 7.62–7.84 (m, 4 H), 8.35–8.43 (m, 2 H), 8.75–8.77 (m, 1 H), 10.49 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 20.4, 120.4, 120.5, 122.3, 126.0, 129.0, 130.1, 133.1, 134.0, 147.7, 162.9 ppm. $C_{14}H_{12}N_2O_3$ (256.26): calcd. C 65.62, H 4.72, N 10.93; found C 65.55, H 4.67, N 10.88.

4-Nitro-*N*-(*p*-tolyl)benzamide (5f):[26] Yield 70 % (179 mg). CAS No. 582–78–5. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 2.26 (s, 3 H), 7.15 (d, *J* = 8.0 Hz, 2 H), 7.62–7.65 (m, 2 H), 8.13–8.16 (m, 2 H), 8.34 (d, *J* = 8.7 Hz, 2 H), 10.45 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 20.4, 120.3, 120.4, 123.5, 129.0, 129.1, 133.1, 136.0, 136.1, 140.6, 149.0, 163.5 ppm. $C_{14}H_{12}N_2O_3$ (256.26): calcd. C 65.62, H 4.72, N 10.93; found C 65.57, H 4.67, N 10.88.

***N*-(*tert*-Butyl)-4-nitrobenzamide (5g):**[28] Yield 80 % (177 mg). CAS No. 42498–30–6. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.37 (s, 9 H), 7.96–8.02 (m, 2 H), 8.11 (s, 1 H), 8.22–8.27 (m, 2 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 28.3, 123.2, 128.8, 141.5, 148.6, 164.6 ppm. $C_{11}H_{14}N_2O_3$ (222.24): calcd. C 59.45, H 6.35, N 12.61; found C 59.38, H 6.31, N 12.55.

***N*-Isopropyl-3-nitrobenzamide (5h):** Yield 82 % (171 mg). CAS No. 50445–53–9. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.13 (s, 3 H), 1.15 (s, 3 H), 4.04–4.12 (m, 1 H), 7.66–7.72 (m, 1 H), 8.24–8.30 (m, 2 H), 8.59–8.64 (m, 2 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 22.0, 41.3, 121.8, 125.4, 129.7, 133.6, 136.0, 147.5 ppm. $C_{10}H_{12}N_2O_3$ (208.22): calcd. C 57.69, H 5.81, N 13.45; found C 57.61, H 5.76, N 13.40.

***N*-Ethylbenzamide (5i):**[29] Yield 88 % (131 mg). CAS No. 614–17–5. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.10–1.13 (m, 3 H), 2.78–2.84 (m, 2 H), 7.22–7.34 (m, 3 H), 7.84–7.88 (m, 2 H), 8.74 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 12.3, 34.2, 127.0, 128.1, 131.0, 134.1, 166.5 ppm. $C_9H_{11}NO$ (149.19): calcd. C 72.46, H 7.43, N 9.39; found C 72.41, H 7.37, N 9.32.

2-Phenylacetamide (6a): Yield 90 % (121.6 mg). CAS No. 103–81–1. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 3.35 (s, 2 H), 6.87 (s, 1 H), 7.16–7.31 (m, 5 H), 7.46 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 42.2, 126.2, 128.1, 129.0, 136.4, 172.2 ppm. C_8H_9NO (135.17): calcd. C 71.09, H 6.71, N 10.36; found C 71.00, H 6.64, N 10.31.

***N*-Ethyl-2-phenylacetamide (6b):** Yield 91 % (148 mg). CAS No. 5465–00–9. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.11 (s, 3 H), 2.73–2.76 (m, 2 H), 3.42 (s, 2 H), 7.09–7.26 (m, 5 H), 8.45 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 12.3, 34.1, 42.9, 125.9, 128.0, 128.1, 128.8, 129.0, 136.4, 174.7 ppm. $C_{10}H_{13}NO$ (163.22): calcd. C 73.59, H 8.03, N 8.58; found C 73.51, H 7.98, N 8.52.

***N*-Isopropyl-2-phenylacetamide (6c):** Yield 90 % (159 mg). ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.14–1.18 (m, 6 H), 3.18–3.20 (m, 1 H), 3.34–3.52 (m, 2 H), 7.10–7.29 (m, 5 H), 8.17 (br. s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 20.2, 40.7, 42.9, 126.5, 128.1, 129.2, 134.9, 169.2 ppm. $C_{11}H_{15}NO$ (177.25): calcd. C 74.54, H 8.53, N 7.90; found C 74.48, H 8.47, N 7.85.

***N*-(*tert*-Butyl)-2-phenylacetamide (6d):** Yield 84 % (161 mg). CAS No. 6941–21–5. ^{1}H NMR (250 MHz, $CDCl_3$/TMS): δ = 1.23 (s, 9 H), 3.28–3.34 (m, 2 H), 7.11–7.27 (m, 4 H), 7.61–7.65 (m, 1 H) ppm. ^{13}C

NMR (62.5 MHz, $CDCl_3$/TMS): δ = 28.4, 42.9, 50.0, 126.0, 128.0, 128.8, 136.9, 169.5 ppm. $C_{12}H_{17}NO$ (191.27): calcd. C 75.35, H 8.96, N 7.32; found C 75.27, H 8.91, N 7.25.

2-Phenyl-*N*-(*p*-tolyl)acetamide (6e): Yield 72 % (162 mg). ^1H NMR (250 MHz, $CDCl_3$/TMS): δ = 2.21 (s, 3 H), 3.59 (s, 2 H), 7.06 (d, J = 7.5 Hz, 2 H), 7.22–7.32 (m, 4 H), 7.44–7.47 (m, 2 H), 10.04 (s, 1 H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$/TMS): δ = 20.3, 43.2, 118.9, 119.0, 126.4, 128.18, 128.21, 129.0, 132.0, 136.0, 136.6, 168.8 ppm. $C_{15}H_{15}NO$ (225.29): calcd. C 79.97, H 6.71, N 6.22; found C 79.91, H 6.64, N 6.17.

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