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Benzimidates as *gem*-Diamidation and Amidoindolyzation Cascade Synthons with a Hydrated Ni^{II} Catalyst

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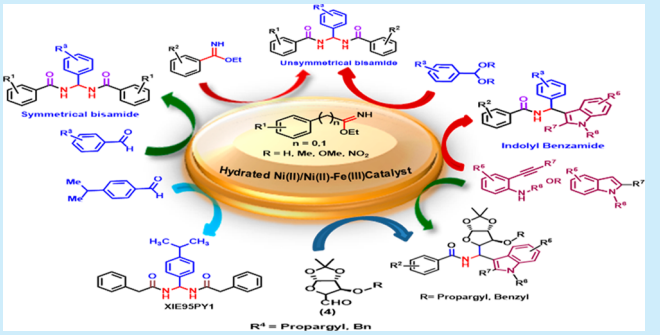
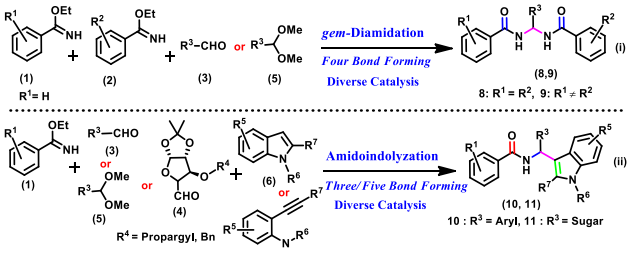
ABSTRACT: We contributed a new benzimidate chemistry through moisture-insensitive Ni^{II}/Ni^{II}-Fe^{III} combo-catalysis for a simultaneous 2−3 bond-forming *gem*-diamidation and amidoindolyzation cascade reaction to construct symmetrical and unsymmetrical *gem*-(arylmethylene)amides and indolo(arylmethylene)-amides, using emerging benzimidate synthons. The operational simplicity, mild nature, generality, and robustness of the strategy were validated through syntheses of a wide range of new molecules, labile sugar-based chiral compounds, and pharmaceuticals with high yields under the same reaction conditions.



Benzimidate[1] is one of the immerging synthons, which may be efficiently utilized in the synthesis of a wide range of compounds to fulfill the growing demands of our modern society. Recently, *o*-C−*H* bonds of benzimidates are activated and functionalized to achieve a wide range of useful compounds.[2] We envisaged a diverse catalysis for simultaneous 2−3 bond-forming cross-coupling of benzimidate cascade synthons (**1** and/or **2**; see Scheme 1) with suitable electron-deficient substrates such as aldehydes (**3**), sugar-based aldehydes (**4**), acetals (**5**), indoles (**6**), and 2-alkynylanilines (**7**) to construct valuable symmetrical and unsymmetrical (arylmethylene)diamides, (indolo)arylmethyleneamides, and sugar-based chiral analogues and pharmaceutical.

Scheme 1. Designed *gem*-Diamidation and Amidoindolyzation Reaction



The symmetrical and unsymmetrical *N*,*N*′-alkylidene bisamides are the key structural motifs of peptidomimetics,[3,4] bioactive natural products including anticancer and antidepressant Leuconoxine (B),[4b] Scholarisine G (A),[4b] antioxidant and antimicrobial JBIR-94 (C),[4a] CB2 receptor inverse agonist, and osteoclast inhibitor XIE95PY1[4c,d] (D, Figure 1), and high blood pressure drugs.[4e] The indolylarylamides are found as

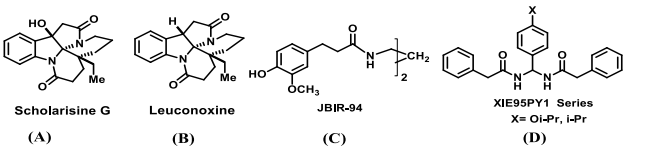


Figure 1. Selected medicinally important*gem*-bisamides.

NOD2 gene mutant, potent melatonin receptor, active agonists and antagonists, antibiotics, antiviral agents, and transcription factor NF-κB inhibitors.[5]

Tremendous application of the diamides has led to the development of many methods, especially in the pharmaceutical industries, using sulfuric acid,[6j] hydrochloric acid,[6i] acetic acid,[6k] trifluoromethanesulfonic acid (CF_3SO_3H),[6h] sulfamic acid (NH_2SO_3H),[6f] *p*-toluenesulfonic acid (*p*-TSA),[6e] boric acid [$B(OH)_3$],[6c] tris(hydrogensulfato)boron [$B(HSO_4)_3$],[6b] phosphotungstic acid,[6d] $H_{14}[NaP_5W_{29}MoO_{110}]$,[6a] fluoroalkanesulfonic acids,[6g] silica-supported SiO_2−$BaCl_2$,[7c] SiO_2−$HClO_4$,[7b] SiO_2−PPA,[7a] and SiO_2−$MgCl_2$.[7d] Nano-copper ferrite ($CuFe_2O_4$),[8a] nano-$SnCl_4$,[8b] $ZnCl_2$,[8c] activated dime-

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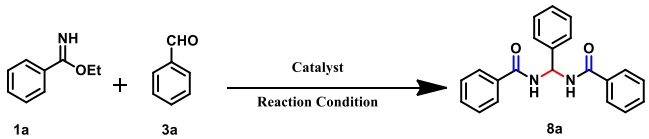
Table 1. Development of Reaction Conditions for *gem*-Diamidation[a]



entry	catalyst	amount (mol %)	solvent	conditions	time (h)	yield[b] (%)
1	$Zn(OTf)_2$	10	toluene	reflux	24	NR[c]
2	$FeCl_3$	10	toluene	reflux	24	NR[c]
3	$Cu(OTf)_2$	10	toluene	reflux	24	NR[c]
4	Ag(OTf)	10	toluene	reflux	24	NR[c]
5	$Ni(OTf)_2$	10	toluene	reflux	12	50[d]
6	$NiCl_2{\cdot}4H_2O$	10	toluene	reflux	10	38[d]
7	$Ru(p\text{-cymene})_2Cl_2$	10	toluene	reflux	8	59[d]
8	$Ni(acac)_2{\cdot}2H_2O$	10	toluene	reflux	10	50[d]
9	**$Ni(OAc)_2{\cdot}4H_2O$**	**10**	**toluene**	**reflux**	**4**	**75**
10	$Ni(OAc)_2{\cdot}4H_2O$	10	benzene	reflux	12	40[d]
11	$Ni(OAc)_2{\cdot}4H_2O$	10	THF	reflux	12	NR[c]
12	$Ni(OAc)_2{\cdot}4H_2O$	10	dioxane	reflux	12	50[d]
13	$Ni(OAc)_2{\cdot}4H_2O$	10	DCE	reflux	12	NR[c]
14	$Ni(OAc)_2{\cdot}4H_2O$	10	DCM	reflux	12	NR[c]
15	$Ni(OAc)_2{\cdot}4H_2O$	10	DMF	100 °C	12	40[d]
16	$Ni(OAc)_2{\cdot}4H_2O$	10	EtOH	reflux	12	NR[c]
17	$Ni(OAc)_2{\cdot}4H_2O$	7	toluene	110	12	52
18	$Ni(OAc)_2{\cdot}4H_2O$	15	toluene	110	4	71
19	**$Ni(OAc)_2{\cdot}4H_2O$**	**10**	**toluene**	**110**	**6**	**74[e]**

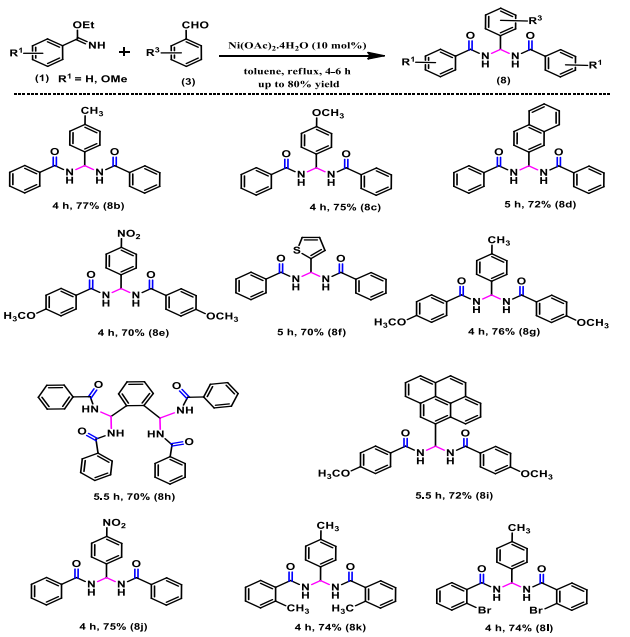
[a]Reactions were conducted using ethylbenzimidate (**1a**, 1 mmol) and benzaldehyde (**3a**, 0.5 mmol) in the presence of catalyst (0.1 mmol). [b]Yield of the isolated product after column purification. [c]No reaction. [d]Unreacted **1a** and **3a** were recovered. [e]Gram-scale synthesis.

thylsulfoxide (DMSO),[8d] and dimethylformamide (DMF)−chlorotrimethylsilane,[8f] as well as the Dean−Stark water trapping approach, have also been utilized, to avoid moisture sensitivity.[8e] There are only limited reports on the synthesis of indolylarylamides utilizing especially designed substrates such as amidoalkylation of indoles, sequential one-pot addition, and aza-Friedel−Crafts reaction.[9] To the best of our knowledge, the diverse catalysis using benzimidates to *gem*-bisamides and indolylarylmethanamides is unknown in the literature. An operationally simple, moisture-insensitive, and general synthetic strategy is highly desirable to access all varieties of new molecules. The generality and mild nature of the reaction may be examined through the construction of labile sugar-based chiral analogues and drug candidates (see Figure 1).

Initially, we examined the formation of the *gem*-bisamide (**8a**), employing ethylbenzimidate (**1a**) and benzaldehyde (**3a**) in the presence of several potential catalysts (10 mol %), such as $Zn(OTf)_2$, $FeCl_3$, $Cu(OTf)_2$, and AgOTf (Table 1, entries 1−4) under refluxing toluene. To our delight, $Ni(OTf)_2$, $NiCl_2$, $Ru(p\text{-cymene})_2Cl_2$, and $Ni(OAc)_2$ promoted the simultaneous two bond-forming couplings to afford the desired **8a** (38%−59%, 8−12 h; see Table 1, entries 5−8). The yield was significantly improved (75%) by hydrated nickel acetate $Ni(OAc)_2{\cdot}4H_2O$ with shorter reaction time (4 h; see Table 1, entry 6). Our solvent survey using nonpolar, polar, and protic media (Table 1, entries 10−16) showed that refluxing toluene is the best choice (Table 1, entry 9). Upon reducing the catalyst loading (7 mol %), **8a** was formed with lower yield (52%; see Table 1, entry 17), while, upon increasing the catalyst loading (15 mol %), a detrimental effect was observed (61%; see Table 1, entry 18). A gram-scale synthesis under standard reaction conditions was successfully verified, affording the desired product with good yield (74%) in 6 h (Table 1, entry 19). Thus, this strategy may find application in both academia and industry.

We proceeded to scrutinize the substrate scope of the reaction utilizing the best reaction conditions (Table 1, entry 9). The reaction of benzimidate (**1a**) with aromatic aldehydes (**3b**−**3d**) afforded the desired *gem*-bisamides (**8b**−**8d**) with high yields (72%−77%, Scheme 2) within 4−5 h. Highly electron-rich *p*-anisaldehyde (**3c**) and deficient *p*-nitrobenzal-

Scheme 2. Synthesized Symmetric *gem*-Bisamides



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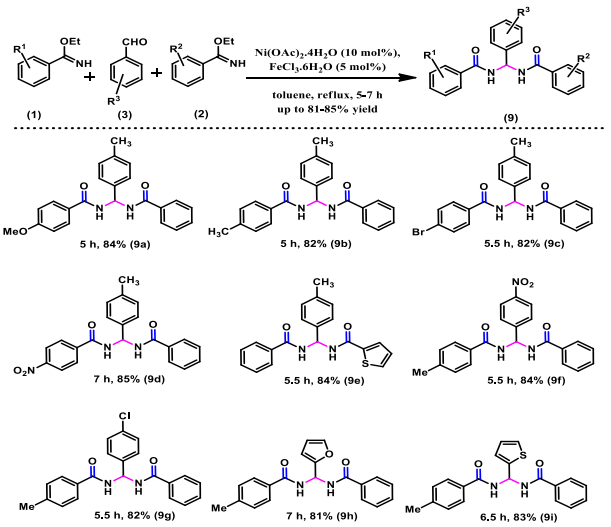
dehyde (**3e**) were examined successfully to produce **8c** and **8e** with a negligible variation in yield (70%−75%). Aromatic hydrocarbon moieties carrying densely π-electron, such as naphthyl and pyrene, as well as heteroaromatic thiophene, were tolerated to produce corresponding **8d**, **8i**, and **8f**, respectively. Dialdehyde compound *o*-phthalaldehyde (**3g**) served as a good substrate to furnish highly sterically hindered *bis*-biamide **8h** in good yield. Gratifyingly, the electronically rich benzimidates (**2a**) were compatible with electronically poor, rich, and densely π-clouded skeleton bearing aldehydes to obtain the desired functionalized bisamides **8e**, **8g**, and **8i**, respectively. Next, we have studied the reaction of benzimidate (**1a**) and electron-deficient *p*-nitrobenzaldehyde (**3e**), which were examined successfully to produce (**8j**) with good yield. Thus, the new reaction is not influenced by the presence of a strongly electron-donating functional group (−OMe, **8c**), as well as an electron-deficient functional group ($-NO_2$, **8j**), present in aldehydes. To our delight, the reactions of sterically hindered ortho-substituted benzimidates (**1b**, **1c**) smoothly produced correspondingly crowded *bis*-amide **8k** and **8l**, respectively. Note that the newly developed simultaneous two-bond-forming *gem*-diamidation reaction is independent of any electronic or steric influence. The structure was established by comparing spectroscopic data of known compound **8a** (see the Supporting Information).

Direct synthesis of dissymmetric *gem*-bisamides is challenging, and it was performed through cross-coupling between differently substituted benzimidates (**2a-e**) and benzimidate (**1**) in 1:1 molar ratio (Scheme 3). The reaction of *p*-

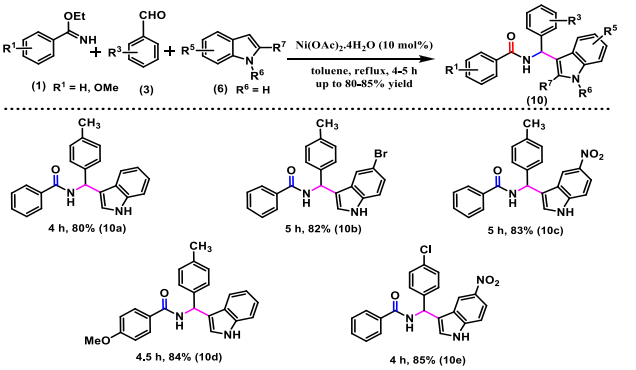
Scheme 3. Dissymmetric Cross-Coupling of Benzimidates



tolylaldehyde (**3b**) with a mixture of benzimidate **2a** and **1** successfully produced **9a** under optimized reaction conditions, using $FeCl_3{\cdot}6H_2O$ (5 mol %) as the cocatalyst in excellent yield (82%). The efficiency of the combo-catalyst[10b] Ni$(OAc)_2{\cdot}4H_2O$ (10 mol %)−$FeCl_3{\cdot}6H_2O$ (5 mol %) was verified through the construction of all new unsymmetrical bisamides **9b**−**9i** with 81%−85% yields. The formation of possible self-coupled products was found in a negligible amount (<5%), and corresponding *N*-benzoylbenzamide byproducts were not detected. Interestingly, the hydrated form of the cocatalyst was more efficient than the anhydrous $FeCl_3$.[10a]

Next, we envisaged direct synthesis of another valuable compound, indolyl(arylmethylene)amides, developing a three-component coupling of benzimidates, indoles, and aldehydes (Scheme 4). The cross-coupling reaction of *p*-tolylaldehyde

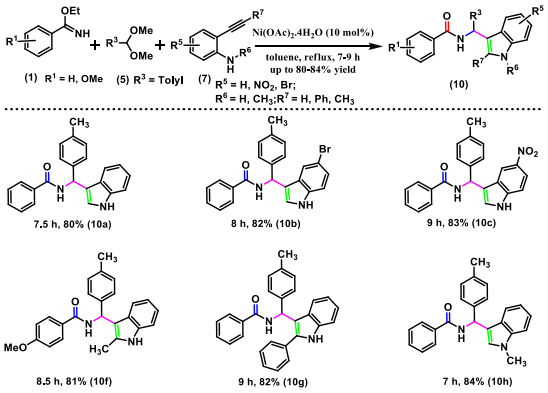
Scheme 4. Direct Synthesis of Indolylbenzamides



(**3b**) or 4-chlorobenzaldehyde (**3i**) with a benzimidate (**1** or **2a**), and electronically different indole derivatives (**6a**−**6e**) afforded the corresponding functionalized 3-indolylarylmethanamides (**10a**−**10e**) in 4−5 h with excellent yield (80%−85%) without any change of conditions (Table 1, entry 6). The synthesized new molecules are potential drug candidates.[5]

We examined the versatility and robustness of the amidoindolyzation strategy through in situ annulation, using 2-alkynyl anilines as a possible precursor to furnish functional groups decorated valuable indolylbenzamides (**10**; see Scheme 5). The reaction between benzimidate (**1a**), mask aldehyde-

Scheme 5. Scope of 2-Alkynylanilines to Indolylbenzamides



acetal (**5a**), and 2-alkynyl anilines (**7**) under standard conditions produced the desired indolylbenzamides (**10a**−**10c**, **10f**−**10h**) with high yield (80%−84%). The novel three-bond-forming cascade reaction is expected to pass through activation of acetal (**5**) by the catalyst, which subsequently proceeds through C−C coupled indolyzation as well as N−C bond forming amidation with benzimidates, and vice versa. We validated the mild nature of the straightforward strategy through the synthesis of carbohydrate-based chiral indolylbenzamides (**11a**, **11b**), using respective pentose sugar aldehyde (**4a**, **4b**) in good yield (70%−72%; see Scheme 6).

The robustness of the method is validated through the direct synthesis of CB2 receptor inverse agonist and osteoclast inhibitor XIE95PY1[4c,d] (**13**; see Scheme 7) by using ethyl 2-phenylacetimidate (**12**) and cumene aldehyde (**3k**) under

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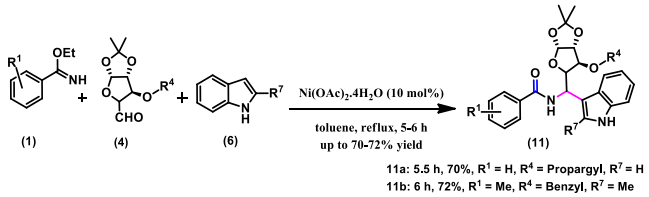
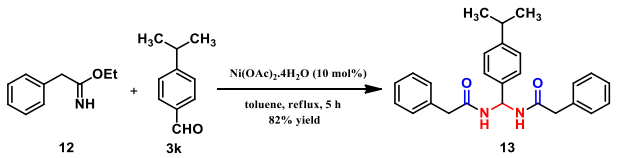
Org. Lett. XXXX, XXX, XXX−XXX

Scheme 6. Synthesized Sugar-Based Chiral Indolylbenzamides



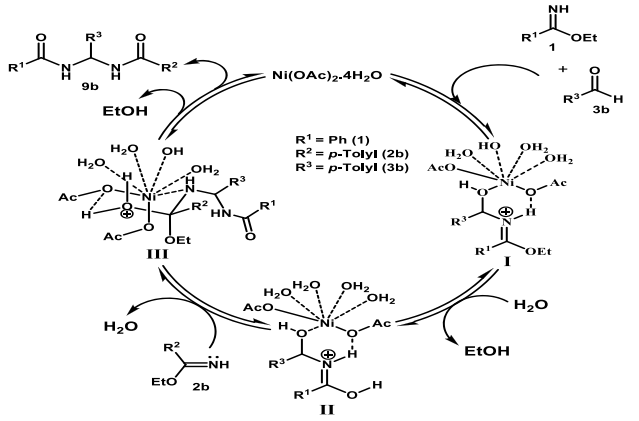
Scheme 7. Synthesis of Bioacive Molecule XIE95PY1



same conditions. This target synthesis further opens up its general feature as the new reaction is also feasible for both benzimidate (**1** and **2**) and acetimidate (**12**) synthons.

It is expected that Ni^{II} catalyst first combines benzimidate and aldehyde/acetal in a stable six-membered intermediate (**I**, Scheme 8), and transformed to **II** through the incorporation of

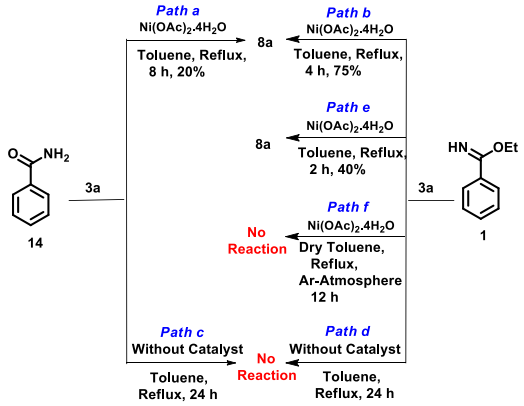
Scheme 8. Proposed Catalysis under Aqueous Surroundings



water and release of ethanol. Interestingly, the formation of EtOH is detected in our ^1H NMR experiment of the ongoing reaction (see the Supporting Information). However, EtOH is somehow associated with other molecules, which is evidenced by observing a higher chemical shift (δ 4.7). The downfield shift (δ 4.0) of CH_3CH_2O- during the start of the reaction may be explained because of the formation of electron-deficient intermediate **I**. Insertion of the other benzimidate and water to the intermediate **II** afforded the cross-coupled putative intermediate **III**, which, upon the release of product *gem*-biamides (**9**), regenerates the catalyst for the next cycle. The active role of the cocatalyst is expected during selective coupling of **2b** to form **III** and/or release of **9b**. However, the exact mechanism is unknown to us; several experiments must be pursued to establish the pathway.

The most important issue of the diverse catalysis is the insensitive nature of the cascade reaction to moisture, which is a major concern for the reported methods. The presence of water is inevitable in the coordination sphere of the Ni^{II} catalyst, as shown in the proposed mechanism. Our control experiments (Scheme 9, path a/b) revealed that benzamide

Scheme 9. Control Experiments



(**14**) and benzimidate (**1**) reacted separately with **3a** to obtain **8a** with 20% and 75% yield, respectively. However, **8a** could not be obtained at all without the catalyst, even after prolonged reaction time (Scheme 9, path c/d). It is an indication of forming transition state **I** (Scheme 8) involving the Ni^{II} catalyst. It was further supported by close monitoring of an ongoing reaction after 2 h, when **8a** was obtained with 40% yield, along with precursor **1** and **3a** (Scheme 9, path e). It supports the formation of no benzamide (**14**). The involvement of water in the coordination sphere of the catalytic system to execute the diverse catalysis is true because no reaction was observed if the reaction is performed under anhydrous conditions (Scheme 9, path f). ESI-MS analyses of an ongoing cross-coupling reaction (R^1 = Ph, R^2 = Tol, R^3 = Tol) revealed the existence of all three predicted intermediates (**I**, *m*/*z* 519.1683; **II**, *m*/*z* 491.2959; **III**, *m*/*z* 654.2631), along with the desired product and precursors.

In conclusion, we report a diverse, general, and moisture-insensitive Ni^{II} catalysis for simultaneous 2−3 bond-forming C−C/C-N/C−O coupling of bezimidate cascade synthons for the unique *gem*-diamidation and amidoindolyzation reactions, which furnished a wide range of new symmetrical, as well as unsymmetrical *gem*-(arylmethylene)dibenzamides, indolyl-(arylmethylene)benzamides, sugar-based chiral analogues, and pharmaceutical utilizing simple substrates. The 2−3 bond-forming reactions with the emerging benzimidate or acetamidate synthons are unknown in the literature. This discovery will open up a new avenue for developing new reactions and chemistry, and synthesizing novel molecules for modern chemical, medicinal, and material sciences.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c00928.

Experimental procedures, characterization data, and ^1H and ^{13}C NMR spectra for all compounds (PDF)

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Notes

The authors declare no competing financial interest.

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