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Construction of indolizine scaffolds from α,ω-alkynoic acids and α,ω-vinylamines *via* sequential-relay catalysis in "one pot"†

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A simple and efficient method has been developed for the synthesis of a diverse range of aryl-fused indolizin-3-ones through sequential Au(I)-catalyzed hydrocarboxylation, aminolysis, and cyclization, followed by ruthenium-catalyzed ring-closing metathesis. Moderate to good yields were observed with satisfactory substrate scope and functional group tolerance. The developed protocol represents a practical strategy for the construction of bioactive aryl-fused indolizin-3-ones.

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Introduction

Nitrogen-containing heterocycles are prevalent structural motifs present in a large number of pharmaceuticals, bioactive compounds and natural products.[1] Functionalized indolizines, particularly aryl-fused ones, broadly exist in a variety of natural and artificial compounds with diverse bioactivities (Fig. 1), representing a class of fundamentally important scaffolds.[2] As useful precursors of indolizine scaffolds and versatile synthons,[3] aryl-fused indolizin-3-ones have aroused significant interest among organic and medicinal chemists. Thus, exploring novel methodologies, particularly those guided by the principles of green chemistry,[4] to construct these scaffolds is of prime importance. In this field, the "multicatalysis" protocol[5] undoubtedly deserves merits due to its ability to convert relatively simple starting materials into more complex products and to significantly reduce time, waste and cost of synthetic processes.

Recently, Zhang reported an efficient sequential relay catalysis to construct aryl-fused indolizinones from *gem*-dibromoolefin using K_2CO_3 and Pd[0] to execute debromination and C–H arylation, respectively (Scheme 1a).[6] In 2016, Kim reported a domino catalysis using palladium-catalyzed intramolecular double Heck reactions to construct the 5-membered ring and 6-membered ring sequentially (Scheme 1b).[7] In 2019, Kumar described a relay catalysis in which enamides were formed through copper-catalyzed Sonogashira coupling followed by intramolecular cyclization, and then obtained isoindolo[2,1-*b*]isoquinolin-7(5*H*)-one derivatives *via* a subsequent intramolecular Heck reaction (Scheme 1c).[8] In 2018, Reddy reported a Rh(III)-catalyzed cascade annulation involving the combination of a $[Cp^*RhCl_2]_2$-catalyzed domino catalysis and a base-mediated intramolecular addition of amide nitrogen to aldehyde (Scheme 1d).[9]

In recent years, the bimetallic relay catalysis has become a useful strategy to activate and construct chemical bonds.[10] This kind of catalytic model is a complement to the existing single catalytic technology.[11] α,ω-Alkynoic acids are building blocks widely used to construct heterocyclic compounds through transition metal-catalyzed cascade reactions.[12–26] We have recently disclosed a Ru-catalyzed cascade reaction of α,ω-alkynoic acids and arylethylamines,[26] the result of which inspired us that if we use α,ω-vinylamines instead of arylethylamines, the corresponding enamide intermediates might be able to produce the desired indolizin-3-ones through ring-closing metathesis. We herein describe this multicatalytic reaction for the synthesis of aryl-fused indolizin-3-ones from α,ω-alkynoic acids and α,ω-vinylamines using $Au(PPh_3)Cl$ and Hoveyda–Grubbs II as catalysts.

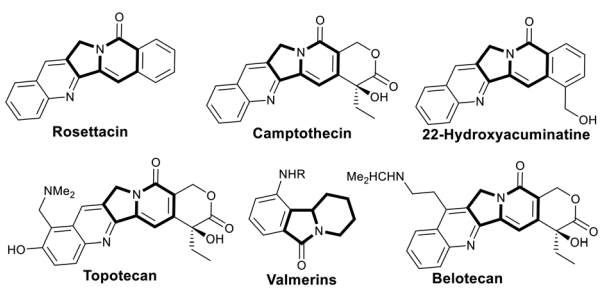

Fig. 1 The representative bioactive molecules.

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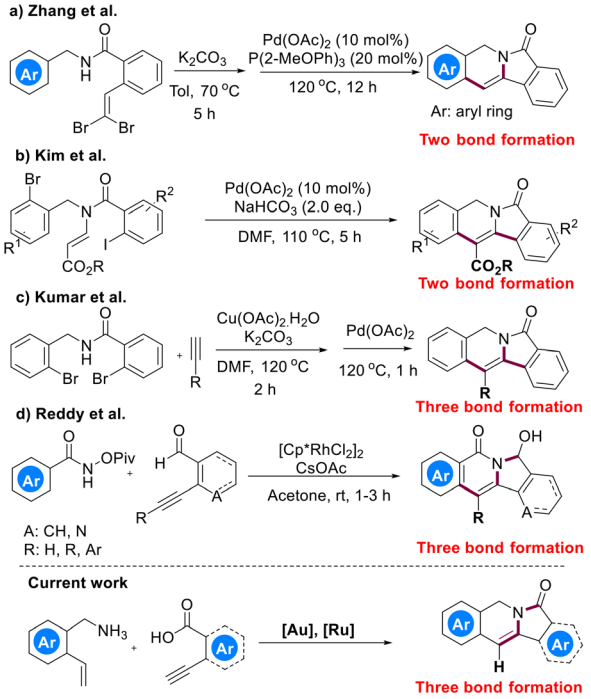


Scheme 1 The representative multicatalysis protocols for the construction of the aryl-fused indolizin-3-ones.

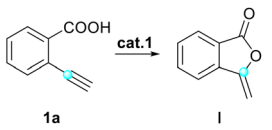
Results and discussion

Our study was guided by the hypothesis that the selected model substrate 2-ethynylbenzoic acid would convert into the desired indolizin-3-one through hydrocarboxylation, aminolysis with 2-vinylbenzylamine, cyclization and ring-closing metathesis. To explore the feasibility of this strategy, we subjected 2-ethynylbenzoic acid (**1a**) and 2-vinylbenzylamine (**2a**) to similar conditions which we used for sequential reactions in our previous work,[26] and used Hoveyda–Grubbs II (**Cat-III**)[27] as the catalyst. After being heated with **Cat-III** for 2 hours in refluxed toluene, **1a** converted into enol lactone (**I**) successfully. Fortunately, we also managed to produce intermediates **II** and **III** with satisfactory yields. (The yields of **I** and **II** were slightly lower than that of **III** due to the volatility of these products and resultant losses during purification.) All the intermediates were separated and elucidated using ^{1}H-NMR, ^{13}C-NMR and mass spectroscopic data. Finally, enamide **III** was heated with **Cat-III** in refluxed toluene and the desired indolizine **3aa** was afforded in 95% isolated yield. This indicated that our strategy to construct indolizin-3-ones is very likely to be viable.

Encouraged by the initial results, we decided to optimize the reaction conditions for the synthesis of **3aa** by varying the catalyst, equivalent and solvent. The results are given in Tables 1–3.

Three commercially available Grubbs' ruthenium carbenes (**Cat-I**, **Cat-II** and **Cat-III**), one representative ruthenium complex (**Cat-IV**) along with a gold catalyst (**Cat-V**) reported for

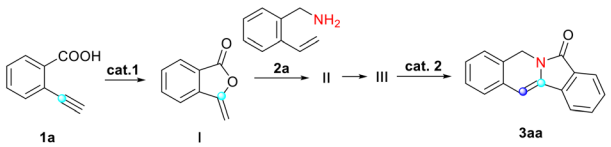
Table 1 Optimization of catalyst 1[a]



Entry	Catalyst 1 (mol%)	Yield (**I**)
1	**Cat-I** (5%)	43%[b]
2	**Cat-II** (5%)	52%[b]
3	**Cat-III** (5%)	90%[b]
4	**Cat-III** (10%)	89%[b]
5	**Cat-IV** (5%)	46%[b]
6	**Cat-V** (5%)	95%[b]
7	**Cat-V** (3%)	94%[b]

[a] Conditions: substrate **1a** (1.0 mmol), anhydrous toluene (10 mL), refluxed for 2 h in a sealed tube. [b] Isolated yield.

Table 2 Optimization of catalyst 2[a]



Entry	Catalyst 1 (mol%)	Catalyst 2 (mol%)	Yield (**3aa**)[b]
1	**Cat-V** (5%)	**Cat-I** (5%)	14%
2		**Cat-II** (5%)	44%
3		**Cat-II** (10%)	46%
4		**Cat-III** (5%)	80%
5		**Cat-III** (10%)	81%

[a] Conditions: substrate **1a** (1.0 mmol), **2a** (1.0 mmol), anhydrous toluene (15 mL), refluxed for 2 h in a sealed tube. [b] Isolated yield.

the hydrocarboxylation of terminal alkynes[16–19,22–24] were used in the catalyst screening study (Fig. 2). All five catalysts were screened for their ability to accelerate the hydrocarboxylation of **1a**. It turned out that 5 mol% of **Cat-V** is the most efficient catalyst among the five for the first step and less quantity (3 mol%) led to no significant variation in the yield of enol lactone **I** (Table 1). 5 mol% of **Cat-III** could also catalyze the transformation effectively, whereas increasing the amount of the catalyst (10 mol%) turned out to be a futile attempt to increase its efficiency (Table 1, entries 3 and 4). With the best candidate for catalyst **1** in hand, we tested the three ruthenium carbenes (**Cat-I**, **Cat-II** and **Cat-III**) for the fourth step, and 5 mol% of **Cat-III** (Hoveyda–Grubbs II) was proved to be the most efficient in catalyzing the ring-closing metathesis (Table 2). The result of catalyst screening enlightened us that we might be able to convert **1a** into the desired indolizine **3aa** in a "one pot" manner with **Cat-III** as the sole catalyst due to its ability to accelerate both the hydrocarboxylation and the ring-closing metathesis. However, this idea was soon proved infeasible, as this domino catalysis protocol with **Cat-III** as the catalyst failed to produce the desired indolizine **3aa**, and increasing the quantity of **Cat-III** at the very beginning of the

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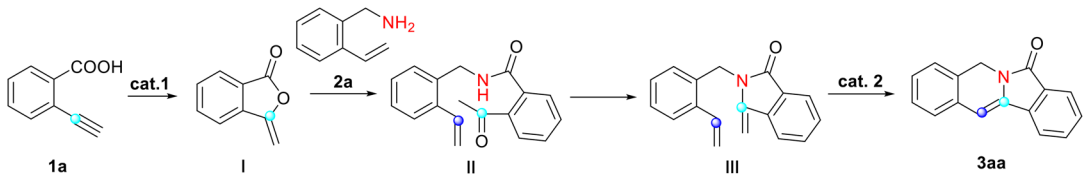
Table 3 Optimized conditions for the model reaction[a]



Entry	Catalyst 1 (mol%)	Catalyst 2 (mol%)	**1a**/**2a** (mmol/mmol)	Solvent	Yield (**I**)	Yield (**III**)[b]	Yield (**3aa**)[b]
1	**Cat-III** (10%)	**Cat-III** (10%)	1.2/1.0	Toluene	—	—	66%
2	**Cat-III** (10%)	**Cat-III** (10%)	1.0/1.0	Toluene	—	—	70%
3	**Cat-III** (10%)	**Cat-III** (10%)	1.0/1.2	Toluene	—	—	68%
4	**Cat-V** (5%)	—	1.0/1.0	Toluene	95%[c]	90%	—
5	**Cat-V** (5%)	—	1.0/1.0	DCM	90%[c]	18%	—
6	**Cat-V** (5%)	—	1.0/1.0	DCE	84%[c]	47%	—

[a] Conditions: substrate **1a** (quantity noted), **2a** (quantity noted), anhydrous solvent (15 mL), refluxed overnight in a sealed tube. [b] Isolated yield. [c] Determined by crude NMR analysis.

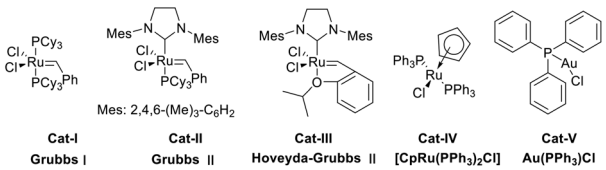
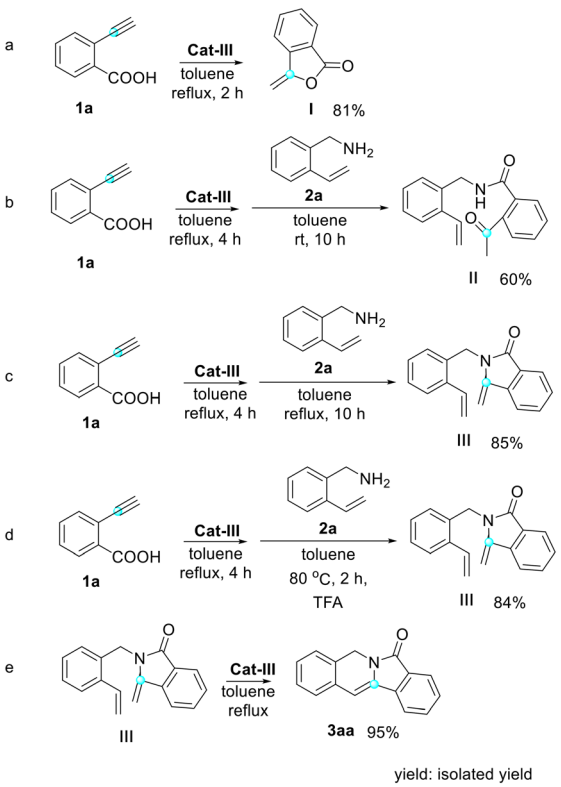


Fig. 2 Catalysts screened in this work.



Scheme 2 The control experiments.

procedure made no difference to the disappointing result. A satisfactory yield of **3aa** could only be achieved by adding fresh **Cat-III** into the reaction medium after the formation of enamide **III**. One probable explanation is that hydrocarboxylation is catalyzed by the decomposed product of **Cat-III** instead of **Cat-III** itself,[28] therefore by the time the intermediate **III** was formed, there was little amount of **Cat-III** existing in the reaction medium and hence, **3aa** could not be afforded in the desired quantity *via* ring-closing metathesis.

An equivalent screening study was then conducted. At the very beginning, we were worried that the remaining 2-vinylbenzylamine **2a** in the reaction medium might hinder the ring-closing metathesis by poisoning the ruthenium catalyst **Cat-III**. To our delight, the result indicated that a slight modification of the **1a**/**2a** ratio has no significant influence on the yield of **3aa** (Table 3, entries 1–3). According to the result of the optimization study, we deemed it wiser to maintain the initial ratio of 1 : 1 to achieve the best yield. We also examined the effect of various solvents and found that the yield of enamide **III** dropped significantly when DCM and DCE were used. The three solvents investigated in this study differed in their boiling points, which might explain why this protocol failed to produce intermediate **III** in the desired quantity in DCE and DCM. Given the fact that enol lactone **I** could be produced smoothly in both DCM and DCE (entries 16–18) and the formation of intermediate **II** did not require high temperature (Scheme 2), we assumed that high temperature might be fundamental for the cyclization of **II** and the low yield of **III** in DCM and DCE might be attributed to the comparatively low boiling points of DCM and DCE. To find proof for this hypothesis, we investigated the influence of reaction temperature in every step of the protocol using toluene as the solvent. The results (shown in Table S1†) further verified our assumption that the formation of **III** requires high temperature and revealed that the optimized temperature for this one pot reaction is 115 °C (temperature set for the oil bath). Nevertheless, we found that for volatile aliphatic alkynoic acids like pent-4-

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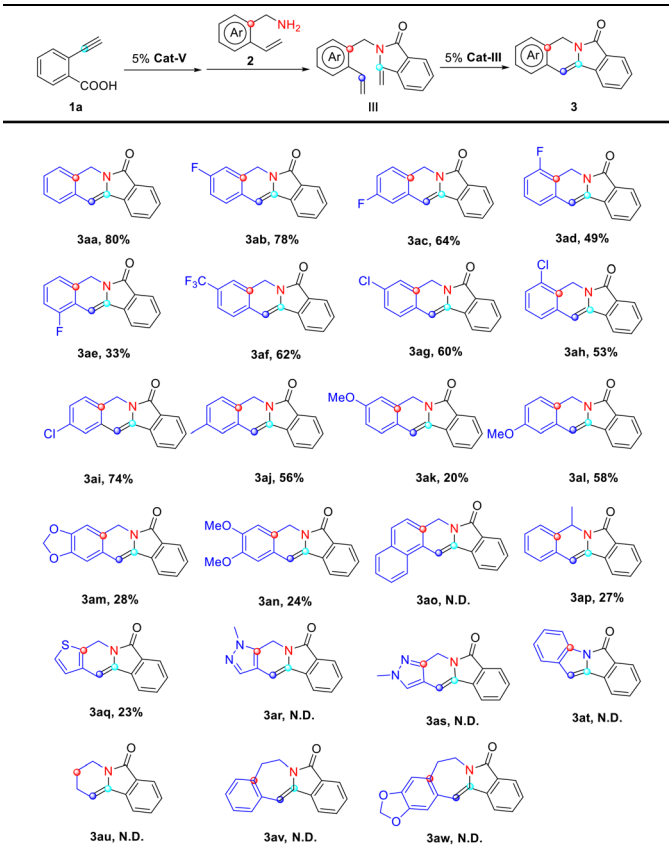
ynoic acid **1j** (see Table S2†), it is difficult to fully convert them into the corresponding enol lactone **I** in refluxed toluene. DCM and DCE might be better solvents than toluene in the hydrocarboxylation step when the α,ω-alkynoic acid substrate is an aliphatic one, since pent-4-ynoic acid produced intermediate **I** in far better yields (95–96%) in DCM and DCE. Thus, for volatile aliphatic alkynoic acid substrates, it is wise to choose DCM or DCE as the solvent at the beginning, remove the solvent after the formation of intermediate **II**, and introduce toluene into the reacting system as it has a higher boiling point.

Now that we have known the cyclization step is the key step of the whole procedure, we tried to optimize the reaction conditions by introducing additives into the catalytic system. Considering the importance of Brønsted or Lewis acids in the cyclization step of this multicatalytic reaction, we tested TFA, a privileged additive reported in similar reactions,[17,19,23] and found that TFA could indeed promote the subsequent transformation (see Table S3†). Other additives such as CH_3COOH and Ac_2O[29] could also lower the temperature required for the formation of enamide **III** to 80 °C, but cannot match the efficiency of TFA. Therefore, we considered TFA as the best additive among all the acids tested. However, for aliphatic alkynoic acid substrates like **1j**, this approach failed to give the corresponding enamide **III** at lower temperatures (see Table S3†). What's more, enamide **III** could not convert into the desired indolizine **3** in the desired quantity at 80 °C (Table S1†). As we intended to accomplish the synthesis of indolizine **3** in a "one pot" manner, we decided not to include TFA to our protocol and to maintain the initial optimized temperature for the cyclization step in order to simplify the catalytic system.

With the optimal reaction conditions in hand, we then examined the reaction of various amines (**2**) with 2-ethynylbenzoic acid (**1a**) to probe the generality of the protocol (Table 4). The performance of different amine substrates diverge from each other dramatically. Generally speaking, electron-withdrawing group-substituted vinylamines gave the corresponding indolizines (**3**) in higher yields than electron-donating group-substituted ones (**3ab**–**3an**). The structure of indolizine **3af** was unambiguously confirmed by single crystal X-ray analysis (Fig. 3). For amines bearing the same substituents, the position of the substitution has a direct impact on the yield of the corresponding product **3**, partly due to the steric hindrance hampering the ring-closing metathesis. A comparison between vinylamine substrate **2b**–**e** indicated that the substitution at the *ortho*-position of the vinyl group is particularly undesirable. Even small substituents like fluorine at that position undermined the yield of the corresponding indolizine **3** dramatically, which offered an explanation to our failure in producing indolizine **3ao** from vinylamine substrate **2o**. Substitutions at other positions of the benzene ring, however, are well-tolerated. We even observed the formation of indolizine **3ap**, indicating that the substitution at the benzyl position could be tolerated as well. We then explored whether this protocol could be applied to heterocyclic amines. To our dismay,

Table 4 The scope of vinylamines in the reaction[a]



[a] Conditions: substrate **1a** (1.0 mmol), **2a**–**2u** (1.0 mmol), **Cat-V** (5 mol%, 25 mg), **Cat-III** (5 mol%, 32 mg), toluene (15 mL), refluxed overnight; yield: isolated yield; N.D. not detected.

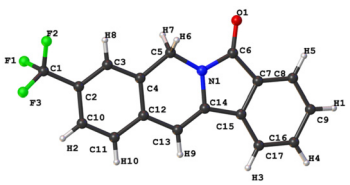


Fig. 3 X-ray crystal structure of **3af** (CCDC no. 2073382†).

electron-rich heterocyclic amines such as **2q** suffered the same fate as electron-donating group-substituted phenylmethanamines, whereas nitrogen-containing heterocyclic amines such as **2r** and **2s** simply failed to give the expected indolizines, probably due to their complexion with the Cat-**III** catalyst. Our attempts to extend the substrate scope of this protocol to aniline (**2t**), phenethylamine (**2v** and **2w**) and aliphatic amine (**2u**) also met with failure. Nevertheless, we observed the formation of enamide intermediate **III** in the case of **2u**–**w**, indicating that ring-closing metathesis catalyzed by Cat-**III** might not be applicable for these enamide substrates under the given conditions.

To further extend the substrate scope of this multicatalytic reaction, a number of diverse alkynoic acids (**1**) were tested

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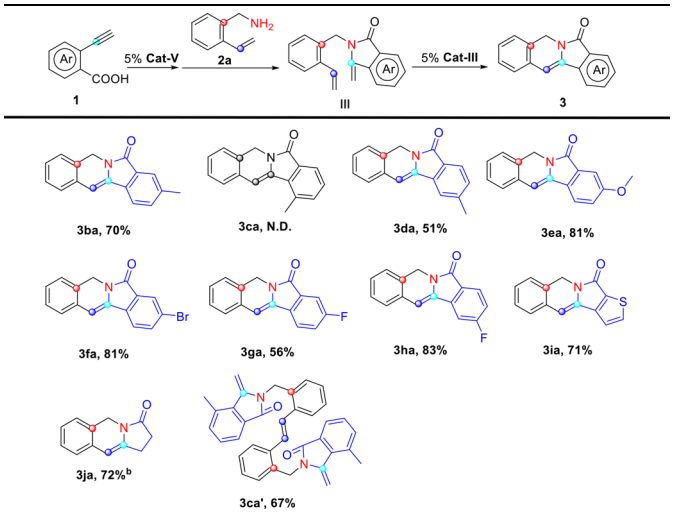
Table 5 The scope of alkynoic acids in the reaction[a]



[a] Conditions: substrate 1 (1.0 mmol), **2a** (1.0 mmol), **Cat-V** (5 mol%, 25 mg), **Cat-III** (5 mol%, 32 mg), toluene (15 mL), refluxed overnight; yield: isolated yield; N.D. not detected. [b] DCE (10 mL) was used as solvent in the hydrocarboxylation step, removed after the formation of intermediate **II** and switched to toluene (10 mL) as solvent afterwards.

(Table 5). To our delight, the substitution on benzoic acid is well-tolerated. Both alkynoic acids bearing electron-donating and electron-withdrawing groups gave indolizine **3** in moderate to good yields (51–83%). The highest yield was observed in the case of fluorine-substituted alkynoic acid **1h** (83%), whereas methoxyl-substituted alkynoic acid **1e** and bromo-substituted alkynoic acid **1f** also gave the corresponding indolizine **3** in a satisfactory yield of over 80%. Even heterocyclic alkynoic acid (**1i**) and aliphatic alkynoic acid (**1j**) reacted smoothly with **2a** to give the expected product in reasonably good yields (71% and 72% yields, respectively). The only exception is alkynoic acid **1c**, whose enamide intermediate formed compound **3ca′** instead of the expected indolizine *via* intermolecular cross metathesis, indicating that an increase of steric hindrance might still be a challenge for ring-closing metathesis in this protocol.

Conclusions

In summary, this study presents a simple, practical and environmentally benign protocol involving Au(I)-catalyzed hydrocarboxylation, aminolysis, cyclization and ruthenium-catalyzed ring-closing metathesis. This unified approach provided indolizin-3-ones in moderate to good yields with satisfying substrate scope and functional group tolerance.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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