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# Palladium-Catalyzed Cascade Decarboxylative Amination/6-*endo*-*dig* Benzannulation of *o*-Alkynylarylketones with *N*-Hydroxyamides To Access Diverse 1-Naphthylamine Derivatives

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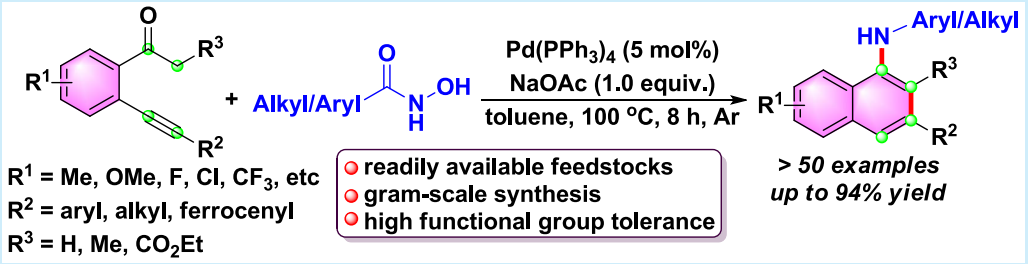
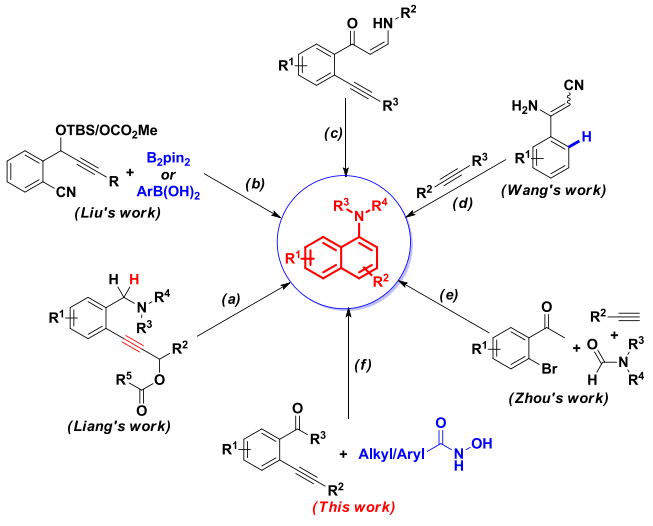



**ABSTRACT:** An efficient and practical one-pot strategy to produce highly substituted 1-naphthylamines via sequential palladium-catalyzed decarboxylative amination/intramolecular 6-*endo*-*dig* benzannulation reactions has been described. In this reaction, a broad range of electron-rich, electron-neutral, and electron-deficient *o*-alkynylarylketones react well with *N*-hydroxyl aryl/alkylamides to give a diversity of 1-naphthylamines in good to excellent yields under mild reaction conditions. The gram-scale synthesis, with benefits such as undiminished product yield and easy transformation, illustrated the practicality of this method.

Compounds incorporating a naphthalene ring structure exist in a broad array of both natural products and bioactive molecules, which have been found to exhibit a wide spectrum of biological activities.[1] Among them, 1-naphthylamines represent an important class that is found in key structural frameworks of numerous biologically active molecules and functional materials.[2] As a consequence, great effort has been devoted to developing efficient protocols for their preparation.[3] Intermolecular and intramolecular benzannulations have attracted considerable attention as the efficient and reliable synthetic tool for the synthesis of 1-naphthylamines in a single operation (Scheme 1a).[4] Very recently, Liu and coworkers developed the copper- or nickel-catalyzed intramolecular benzannulation of *o*-allenylaryl nitriles for the synthesis of 3-boryl/aryl-1-naphthylamine derivatives (Scheme 1b).[5] Li's group reported a $AgNO_3$-catalyzed cycloisomerization of *o*-alkynylphenyl enaminones for the synthesis of 4-acyl-1-naphthylamine derivatives (Scheme 1c).[6] Wang and coworkers disclosed an intermolecular annulation of *β*-enaminonitriles with alkynes via Rh(III)-catalyzed C−H activation to construct highly substituted 1-naphthylamines (Scheme 1d).[7] In 2019, Zhou and coauthors found that amides could be used as aminating agents, which were excellent alternatives to toxic and/or odorous amines, and they developed an efficient, convenient, and general method to directly synthesize the valuable functionalized 1-naphthylamines from readily available terminal alkynes, 2-bromoaryl ketones, and amides via copper-catalyzed benzannulation in a

**Scheme 1. Transition-Metal-Catalyzed Intermolecular and Intramolecular Benzannulations for Construction 1-Naphthylamine Skeleton**

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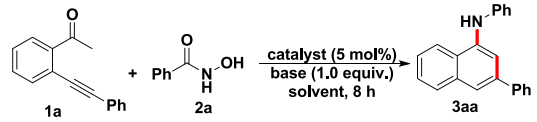
green solvent (Scheme 1e).[8] In addition, copper-catalyzed aminobenzannulation of *ortho*-alkynylarylketones with amines has been described by Wen's and Hua's group, respectively.[9] Consequently, the development of new efficient strategies that provide straightforward access to highly functionalized 1-naphthylamines is of significant importance.

It has been well established that *ortho*-alkynyl aryl carbonyl species are competent reactants endowed with multiple reactive sites, thus they have been widely served as versatile precursors for many important targets of chemical and biomedical potentials, allowing them to undergo a variety of transformations, such as nucleophilic additions and Diels–Alder reactions.[10] As a member of the family of *o*-alkynyl aryl carbonyl compounds, *o*-alkynylarylketones also attract great interest within the scientific community.[11] It is well-known that *o*-alkynyl aryl ketones preferentially undergo the Lewis/Brøsted acid induced intramolecular 5-*exo*-*dig* and 6-*endo*-*dig* oxocyclization to form a variety of biological isobenzofuran isobenzopyran derivatives[12] as well as carbocyclization to prepare the useful 1-indanone and 1-naphthol skeletons.[13] A literature survey indicated that the synthesis of 1-naphthylamine derivatives via the cascade reaction starting from 6-*endo*-*dig* carbocylization using the *o*-alkynylarylketones, to the best of our knowledge, remains rare. Herein, we report the first palladium-catalyzed cascade decarboxylative amination/6-*endo*-*dig* benzannulation of *o*-alkynylarylketones with *N*-hydroxyamides, which provides an efficient protocol for highly functionalized 1-naphthylamine derivatives (Scheme 1f).

Initially, by using the $Pd(OAc)_2/PPh_3$ as a catalyst system, the reaction of 1-(2-(phenylethynyl)phenyl)ethan-1-one (**1a**) and *N*-hydroxybenzamide (**2a**) was performed in the presence of NaOAc as an additive in toluene, and the desired product **3aa** was isolated in 68% yield (Table 1, entry 1). Encouraged by this result, we further examined the reactivity in other palladium catalysts (Table 1, entries 2, 3). To our delight, compound **3aa** was generated in 94% yield when $Pd(PPh_3)_4$ was used as a catalyst in the absence of ligand. Control experiments proved that the presences of $Pd(PPh_3)_4$ and base were both mandatory for the formation of **3aa** (Table 1, entries 4, 5). Next, various common inorganic and/or organic bases were investigated to modulate the product formation (Table 1, entries 5–11), and sodium acetate (NaOAc) was proven to be the best choice. Afterward, the solvent effect was also evaluated (Table 1, entries 12–17), and toluene was finally proven to be the optimal solvent for the formation of compound **3aa**. Further optimization of the reaction conditions indicated that a 5 mol % $Pd(PPh_3)_4$/1.0 equiv of NaOAc catalyst system was sufficient to promote this cascade reaction (Table 1, entry 2).

With the optimized reaction conditions in hand, we then started to investigate the *N*-hydroxyamides for this cascade decarboxylative amination/6-*endo*-*dig* benzannulation process with 1-(2-(phenylethynyl)phenyl)ethan-1-one (**2a**). As shown in Table 2, both alkyl and aryl *N*-hydroxyamides were well proceeded under the optimal conditions, giving the desired products **3** in moderate to excellent yields (68–94%) regardless of their substitution patterns and electronic natures. Electron-rich (4-OMe), electron-neutral (4-Me, 4-$^t$Bu, 3-Me) and electron-deficient (4-F, 4-Cl, 4-Br, 4-$CF_3$, 3-Br) *N*-hydroxyl aryl amides provided the corresponding products (**3aa**–**3am**) in moderate to excellent yields. It was particularly that sterically hindered *N*-hydroxyl arylamides were successfully coupled with **1a** and generated *mono*- and di-*ortho*-substituted *N*-phenylnaphthalen-1-amines in 75–88%yields

**Table 1. Optimization of the Reaction Conditions**[a]




| entry | catalyst | base | solvent | temp/°C | yield/%[b] |
|---|---|---|---|---|---|
| 1[c] | $Pd(OAc)_2$ | NaOAc | toluene | 100 | 68 |
| 2 | $Pd(PPh_3)_4$ | NaOAc | toluene | 100 | 94 |
| 3 | $Pd_2(dba)_3$ | NaOAc | toluene | 100 | 74 |
| 4 | – | NaOAc | toluene | 100 | nr |
| 5 | $Pd(PPh_3)_4$ | – | toluene | 100 | nr |
| 6 | $Pd(PPh_3)_4$ | CsOAc | toluene | 100 | 81 |
| 7 | $Pd(PPh_3)_4$ | AgOAc | toluene | 100 | 85 |
| 8 | $Pd(PPh_3)_4$ | KOAc | toluene | 100 | 87 |
| 9 | $Pd(PPh_3)_4$ | $NaHCO_3$ | toluene | 100 | 54 |
| 10 | $Pd(PPh_3)_4$ | $Na_2CO_3$ | toluene | 100 | 57 |
| 11 | $Pd(PPh_3)_4$ | $K_2CO_3$ | toluene | 100 | 15 |
| 12 | $Pd(PPh_3)_4$ | $Et_3N$ | toluene | 100 | 37 |
| 13 | $Pd(PPh_3)_4$ | NaOAc | PhCl | 100 | 51 |
| 14 | $Pd(PPh_3)_4$ | NaOAc | DMF | 100 | trace |
| 15 | $Pd(PPh_3)_4$ | NaOAc | $MeNO_2$ | 100 | trace |
| 16 | $Pd(PPh_3)_4$ | NaOAc | MeCN | 100 | 29 |
| 17 | $Pd(PPh_3)_4$ | NaOAc | DCE | 100 | 53 |
| 18 | $Pd(PPh_3)_4$ | NaOAc | THF | 100 | 68 |
| 19 | $Pd(PPh_3)_4$ | NaOAc | toluene | reflux | 92 |
| 20 | $Pd(PPh_3)_4$ | NaOAc | toluene | 50 | 48 |
| 21[d] | $Pd(PPh_3)_4$ | NaOAc | toluene | refulx | 48 |
| 22[e] | $Pd(PPh_3)_4$ | NaOAc | toluene | refulx | 92 |
| 23[f] | $Pd(PPh_3)_4$ | NaOAc | toluene | refulx | 41 |
| 24[g] | $Pd(PPh_3)_4$ | NaOAc | toluene | refulx | 86 |
| 25[h] | $Pd(PPh_3)_4$ | NaOAc | toluene | refulx | 48 |
| 26[i] | $Pd(PPh_3)_4$ | NaOAc | toluene | refulx | 94 |
| 27[j] | $Pd(PPh_3)_4$ | NaOAc | toluene | refulx | 70 |

[a]Reaction conditions: 1-(2-(phenylethynyl)phenyl)ethan-1-one **1a** (0.25 mmol), *N*-hydroxybenzamide **2a** (0.3 mmol), solvent (3 mL), catalyst (5 mol %), and base (1.0 equiv) under an argon atmosphere for 8 h. [b]Isolated yields. [c]$PPh_3$ used as ligand. [d]The catalyst loading was 2.5 mol %. [e]The catalyst loading was 10 mol %. [f]The base loading was 0.5 equiv. [g]The base loading was 2.0 equiv. [h]Reaction time for 4 h. [i]Reaction time for 16 h. [j]1 equiv of **2a** was used.

(Table 2, **3aj**, **3al**, **3an**, **3ao**, and **3aq**, respectively). Encouragingly, disubstituted *N*-hydroxybenzamide, *N*-hydroxy-2-phenylacetamide, and *N*-hydroxylnaphthamides were also compatible with this palladium-catalyzed cascade transformation with good yields (Table 2, **3ap**, **3ar**–**3at**). Interestingly, *N*-hydroxyl alkyl amides, such as *t*-butyl, cyclohexyl, and adamantly, gave the desired products **3au**, **3av**, and **3aw** in 85%, 89%, and 76% yields, respectively. Moreover, the structure of compound **3aq** was unambiguously characterized via single crystal X-ray crystallographic analysis (see the Supporting Information, for details).

We subsequently examined the structural diversity of various *o*-alkynylarylketones **2** by assessing the substitution effect on both the two benzene rings and $R^4$ substituent. First, substrates $R^2$ with a broad range of substitution were examined. Substituents including electron-donating groups (–OMe, –Me) and electron-withdrawing groups (–F, –Cl, $-CF_3$) at the 4-, 5-, and 6-positions of the benzene rings were well tolerated, affording the corresponding products **3ba**–**3ma** in good to excellent yields. No significant effect was observed when altering the position of the fluoro group (**3da**, **3ha**, and


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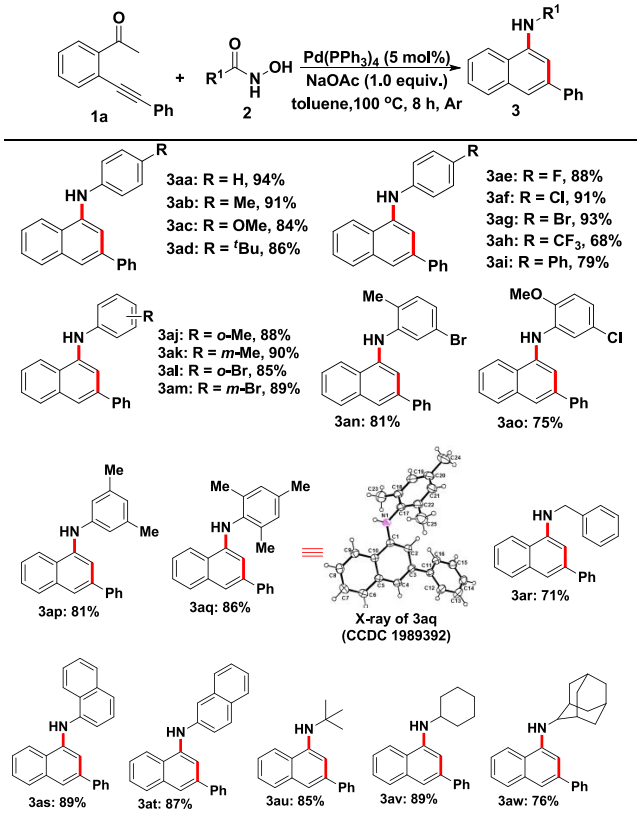
**Table 2. Substrate Scope of *N*-Hydroxyamides 1[a,b]**

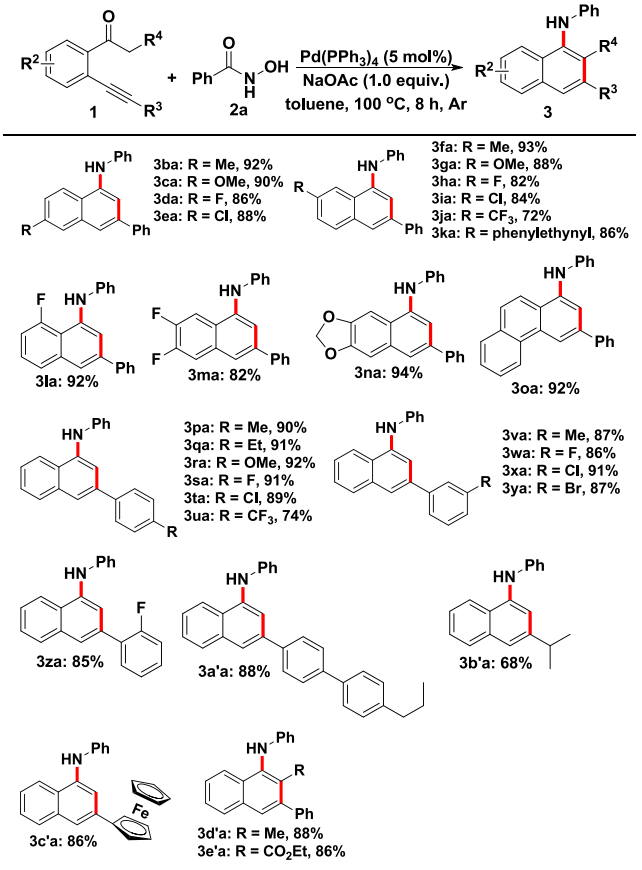



[a]Reaction conditions: unless otherwise specified, 1-(2-(phenylethynyl)phenyl)ethan-1-one **2a** (0.25 mmol), *N*-hydroxyamide **1** (0.3 mmol, 1.2 equiv), $Pd(PPh_3)_4$ (5 mol %), and NaOAc (1.0 equiv) in toluene (3 mL) under an argon atmosphere at 100 °C for 8 h. [b]Isolated yields.

**3la** showed 86%, 82%, and 92% yields, respectively). Reaction with electron-rich *o*-alkynylarylketones smoothly afforded the desired products **3na** and **3oa** in 94% and 92% yields, respectively. The same results were then observed with $R^3$ substituents bearing both electron-donating groups (−Me, −Et, −OMe) and electron-withdrawing groups (−F, −Cl, −Br, $-CF_3$) at the phenyl rings regardless of their substitution positions (Table 3, **3pa**−**3za**). Moreover, the phenyl group could be replaced by isopropyl or ferrocenyl, leading to the desired 1-naphthylamines **3b′a** and **3c′a** in 68% and 86% yields, respectively. Additionally, we turned our attention to studying the suitability of the substituent $R^4$ (changing methyl to ethyl or acetyl ethyl ester group), and the desired products **3d′a** and **3e′a** were successfully obtained in 88% and 86% yields, respectively.

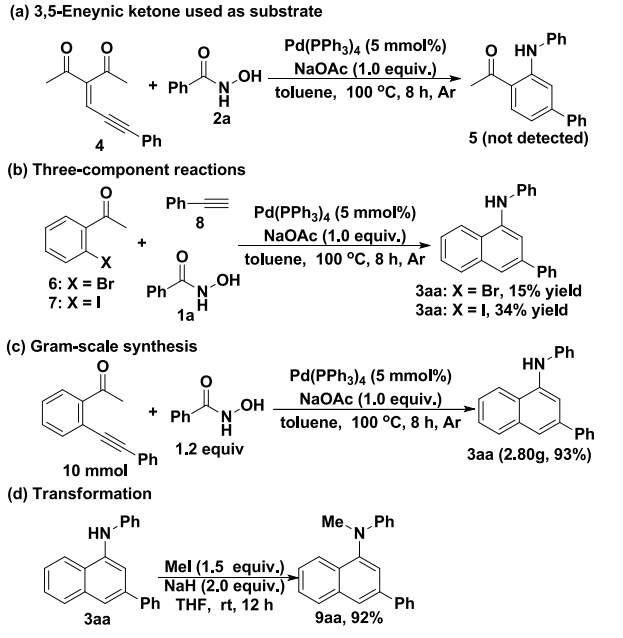
To further amplify the substrate diversity, nonaromatic acetylenone, such as 3,5-eneynic ketone, was deployed for this palladium-catalyzed cascade reaction. The isolated product was the intramolecular 5-*exo*-*dig* oxocyclization rather than the desired diphenylamine **5**, and the isolated yield is 75% yield (Scheme 2a). Next, the feasibility of the palladium-catalyzed three-component reaction of 2-haloaryl ketones (**6**: X = Br and **7**: X = I), phenylacetylene **8**, and *N*-hydroxybenzamide **1a** was investigated, affording the desired product **3aa** in 15% and 34% yields, respectively (Scheme 2b). In addition, the reaction between *o*-alkynylarylketone **1a** with *N*-hydroxyamide **2a** was scaled up to 40 times under the standard conditions. The

**Table 3. Scope of Diverse *o*-Alkynylarylketones for the Palladium-Catalyzed Cascade Reactions[a,b]**




[a]Reaction conditions: unless otherwise specified, (*o*-phenylethynyl)-arylketones **2** (0.25 mmol), *N*-hydroxybenzamide **1a** (0.3 mmol), $Pd(PPh_3)_4$ (5 mmol %), and NaOAc (1.0 equiv) in toluene (3 mL) under argon atmosphere at 100 °C for 8 h. [b]Isolated yield.

**Scheme 2. Further Studies**





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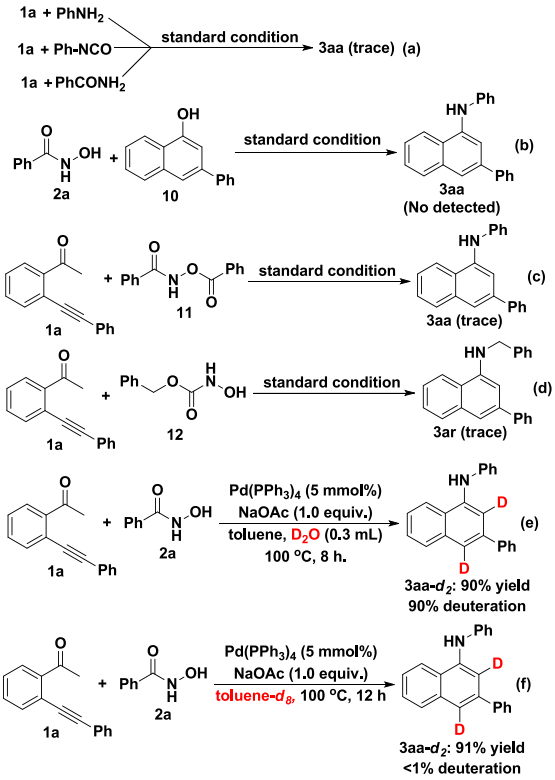
corresponding product **3aa** was afforded on a gram-scale without reducing the percentage yield (Scheme 2c). To showcase the synthetic utilities, product **3aa** was used for late-stage transformation. In the presence of sodium hydride, *N*-methylation between compound **3aa** and iodomethane easily generated *N*,*N*-disubstituted 1-naphthylamine derivative **9aa** in 92% yield (Scheme 2d), highlighting the synthetic utility of the current protocol.

To gain some mechanistic insights into the present protocol, several control experiments were performed (Scheme 3). To

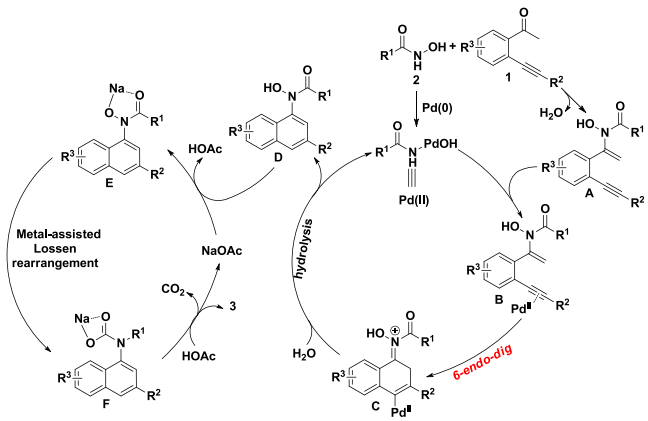
**Scheme 3. Mechanistic Studies**




assess the possibility of key intermediates, aniline, phenyl isocyanate, and benzamide were subjected to the standard condition, respectively (Scheme 3a). Only a trace amount of the desired product **3aa** was produced (detected by TLC). 3-Phenyl-1-naphthalenol **10** was then employed instead of *o*-alkynyl aryl ketone **1a** to investigate whether it was an intermediate in the process, yet the desired product **3aa** was not detected by TLC (Scheme 3b). Furthermore, other amides, such as *N*-(benzoyloxy)benzamide **11** and benzyl hydroxycarbamate **12**, also failed to get the corresponding products under the standard conditions (Scheme 3c, 3d). In addition, a deuterium labeling experiment with the addition of deuterium oxide (0.3 mL) to the reaction system was carried out, and the result indicated only the deuterated product **3aa**-$d_2$ with 90% deuteration at the C-2 and C-4 position was obtained in 90% yield (Scheme 3e). However, the deuterated product was hardly obtained when the reaction was performed in toluene-$d_8$ for12 h (Scheme 3f), illustrating that the reaction may proceed via a keto−enol tautomerization, dehydration, and/or hydrolysis process.

On the basis of the experimental results and literature reports,[8] a plausible decarboxylative amination/intramolecular 6-*endo-dig* benzannulation process was proposed, as depicted in Scheme 4. Initially, the condensation of substrate **1** and **2** to generates the intermediate **A**. Meanwhile, the oxidative

**Scheme 4. Proposed Mechanism**




addition of *N*-hydroxyamide **2** to the Pd(0) catalyst generates the Pd(II) catalyst. Subsequently, the coordination of Pd(II) to the triple bond in the intermediate **A** forms the intermediate **B**, followed by an intramolecular 6-*endo-dig* carbocyclization to afford the intermediate **C**. Then, the hydrolysis of intermediate **C** leads to the intermediate **D** with concomitant regeneration of the Pd(II) catalyst. The deprotonation of intermediate **D** in the presence of sodium acetate as a basic salt generates intermediate **E**. Finally, the Lossen rearrangement process occurs as reported in literature,[14] furnishing the intermediate **F**, which quickly performs decarboxylation and protonolysis to afford the desired product **3**.

In conclusion, we developed a palladium-catalyzed cascade decarboxylative amination/6-*endo-dig* benzannulation of *o*-alkynylarylketones with *N*-hydroxyamides, which provided an efficient and convenient protocol for the synthesis of highly functionalized 1-naphthylamines with a wide range of structural diversity under mild reaction conditions, regardless of their attached substituents. Through the palladium-catalyzed cascade reactions, the 1-naphthylamine framework was efficiently accessed, in which one C−C bond, one C−N, and a new benzene ring were formed simultaneously in a single operation with exclusive 6-*endo-dig* selectivity. The reaction can be easily transformed and scaled up to the gram scale without diminishing the product yield.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c01183.

General experimental procedure, characterization data of the compounds (PDF)

### Accession Codes

CCDC 1989392 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.



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### Notes

The authors declare no competing financial interest.

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