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Pd-Catalyzed One-Pot Two-Step Synthesis of 2-(1*H*-indol-3-yl)-2-phenylindolin-3-ones from 2-Alkynyl Arylazides and Indoles

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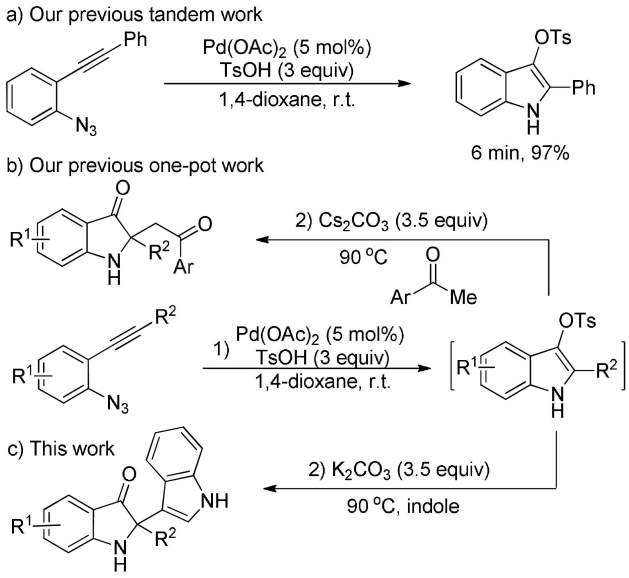
An efficient palladium-catalyzed one-pot two-step reaction of 2-alkynyl arylazides and indoles has been developed. The reaction proceeded well under mild reaction conditions and provided the 2-(1*H*-indole-3-yl)-2-phenylindolin-3-ones in good to excellent yields.This transformation involves a rearrangement of 1*H*-indole-3-sulfonates generated *in situ* and Mannich-type addition of indoline-3-ones.

Introduction

Pseudoindoxyl derivatives[1] are widely found in many bioactive alkaloids,[2] such as Isatisine A,[2a] Trigonoliimine C,[2b] and Cephalinone D.[2c] In particular, the naturally occurring Isatisine A containing indoxyl group at the C-2 position of indolin-3-ones has shown very interesting antiviral properties.[2a] Based on its potential bioactivities, the synthesis of the indolin-3-ones with indoxyl functional group has been paid more attention. Many synthetic methods have been reported,[3] such as trapping metallocarbene by organoazide,[3a] gold/copper co-catalyzed oxidation of 2-alkynylanilines,[3b] TEMPO/Pd-catalyzed oxidation of indoles,[3c] DDQ-promoted oxidative *aza*-Friedel-Crafts alkylation of indole with 3-indolelinone-2-carboxylates,[3d] $InCl_3$-mediated addition of indol-3-one-*N*-oxide,[3e] Pd-catalyzed oxidative dearomatization of 2-arylindoles,[3f] visible light photocatalytic oxygenation of indoles[3g] and Mannich reaction of cyclic-acylimines.[3h–i] Although these reported reactions are efficient methods, the novel strategy for the preparation of indolin-3-ones bearing indoxyl group is still needed.

Recently, construction of heterocycles *via* α-imino metal carbines[4] generated *in situ* from 2-alkynyl arylazides has become an efficient method. In these α-imino metal carbenes formation reactions, gold[5] and palladium[6] were employed by us for the preparation of heterocycles.[7] In 2017, we reported that synthesis of 1*H*-indole-3-sulfonates *via* palladium-catalyzed tandem reactions of 2-alkynyl arylazides with sulfonic acids, which provided the desired products in excellent yields and proceeded very fast (Scheme 1, a).[7b] Recently, we reported



Scheme 1. Palladium-catalyzed one-pot two-step synthesis of 2-(1*H*-indol-3-yl)-2-phenylindolin-3-ones

palladium-catalyzed one-pot synthesis of C2-quaternary indolin-3-ones *via* 1*H*-indole-3-sulfonates Generated *in situ* from 2-alkynyl arylazides and sulfonic Acids. In this work, various aryl ketones were employed as nuclephiles (Scheme 1, b).[7a] Therefore, we envisioned that indoles could be used as good nuclephiles instead of aryl ketones in the one-pot two-step reactions. (Scheme 1, c). As far as we know, there has been no previous report on this transformation of 2-alkynyl arylazides to 2-(1*H*-indol-3-yl)-2-phenylindolin-3-ones *via* one-pot process in organic synthesis. It was noted that the key intermediate 2-aryl-3*H*-indol-3-ones generated *in situ* from simple starting materials were very active and not easily prepared.[8] This one-pot process would also have significant potential as a novel strategy for the total synthesis of Isatisine A. Herein, we disclose a novel palladium-catalyzed one-pot preparation of indolin-3-ones bearing indoxyl group *via* 1*H*-indole-3-sulfonates generated *in situ* from 2-alkynyl arylazides.

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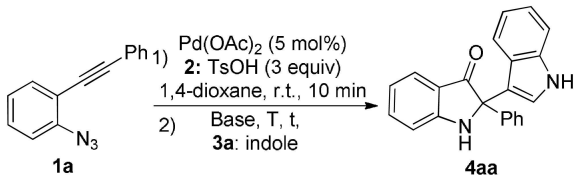
Results and Discussion

At the outset, we chose to focus our attentions on the reaction of 1-azido-2-(phenylethynyl)benzene **1a** and indole **3a** as model substrates to establish the reaction conditions (Table 1).

Table 1. Optimization of reaction conditions.[a]



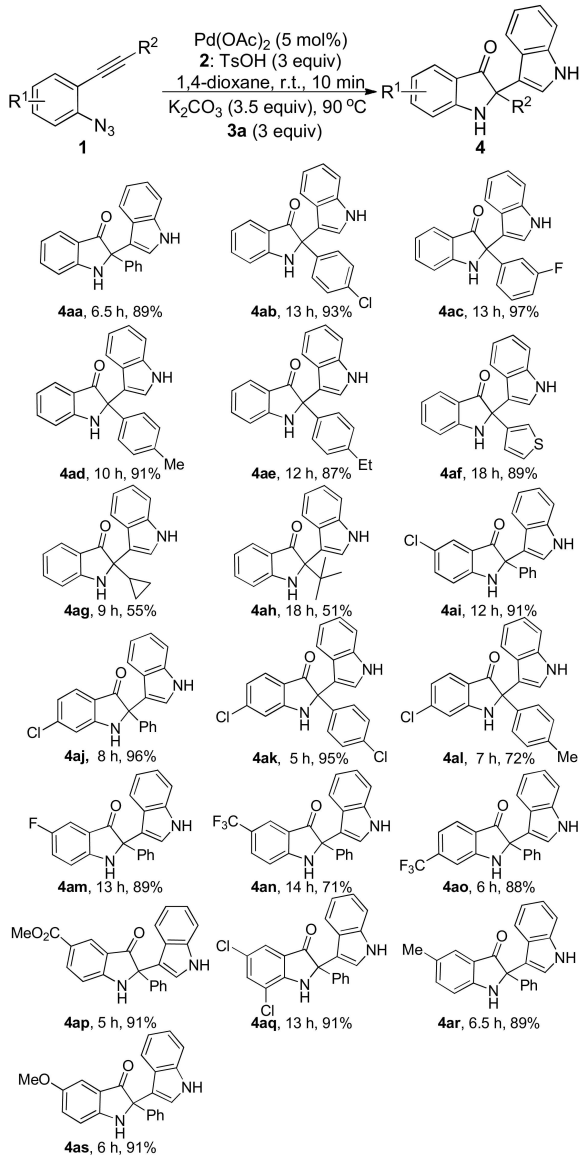
Entry	Base	T (°C)	Time (h)	Yield/%[b]
1	K_2CO_3	90	6	89
2	K_2CO_3	60	6	72
3	Cs_2CO_3	90	6	82
4	K_3PO_4	90	6	61
5	*t*-BuOK	90	6	70
6	DBU	90	2	28
7	-	r.t.	20	n.r.[c,d]
8	-	r.t.	20	n.r.[e,d]
9	K_2CO_3	90	6	86[f]
10	K_2CO_3	90	6	85[g]
11	K_2CO_3	90	6	79[h]

[a] Reaction conditions: **1a** (0.1 mmol), $Pd(OAc)_2$ (5 mol%), **2** (3 equiv), 1,4-dioxane (1 mL), base (3.5 equiv), and **3a** (3 equiv). [b] Isolated yield. [c] DMF was used as solvent. [d] No reaction and **1a** was recovered. [e] DMSO was used as solvent. [f] $Pd(NO_3)_2{\cdot}2H_2O$ was used as catalyst. [g] $PdCl_2$ was used as catalyst. [h] 2 equiv. of indole was used.

The best result was obtained by treatment of 1 equiv. of **1a** and 3 equiv. of TsOH with 5 mol% of $Pd(OAc)_2$ in 1,4-dioxane at room temperature for 10 min and then 3.5 equiv. of K_2CO_3 and 3 equiv. of indole **3a** were added to the reaction solution at 90 °C for 6 h, which furnished **4aa** as the sole product in 89% yield (Table 1, entry 1). In contrast, a lower yield of 72% could be obtained on lowering the reaction temperature to 60 °C (Table 1, entry 2). Notably, various bases were also examined by repeating this reaction. A slightly lower yield of 82% was afforded using Cs_2CO_3 (Table 1, entry 3). On the other hand, when the base was changed to K_3PO_4 and *t*-BuOK, the desired product was obtained in 61% and 70%, respectively (Table 1, entries 4–5). When DBU was used as a base, the reaction proceeded very fast and gave **4aa** in markedly lower yield of 28% (Table 1, entry 6). However, there was no reaction and only the stating material **1a** was recovered when the solvent 1,4-dioxane was changed to DMF or DMSO (Table 1, entries 7–8). Similar yields of 85–86% were also obtained on repeating the reaction with other palladium catalysts such as $Pd(NO_3)_2{\cdot}2H_2O$ and $PdCl_2$ in place of $Pd(OAc)_2$ (Table 1, entries 9–10). The reaction was also checked by using 2 equiv. of indole, and was found to give lower product yield (Table 1, entry 11).

With the optimized reaction conditions in hands, a series of 2-alkynyl arylazides **1** was reacted with various indole derivatives to gain the generality of reaction. Reactions of 2-alkynyl arylazides bearing either an electron-withdrawing or electron-donating group on the alkyne carbon (R^2 = 4-ClPh, 3-FPh, 4-MePh and 4-EtPh) with indole **3a** gave the corresponding 2-(1*H*-indol-3-yl)-2-phenylindolin-3-ones **4** in good to excellent yields (Table 2, **4ab**-**4ae**). 2-Alkynyl arylazide bearing a thiophene moiety was also found to proceed well under the present conditions and gave the corresponding product in 89% yield (Table 2, **4af**). On the other hand, 2-alkynyl arylazides containing an alkyl group were also examined under the standard conditions but were found to give lower yields of 55% and 51%, repectively (Table 2, **4ag-4ah**). Additionally, reactions of starting materials with various substituted group on the aryl ring (R^1 = halide, CF_3, ester, Me and OMe) were

Table 2. Substrate scope to 2-(1*H*-indol-3-yl)-2-phenylindolin-3-ones 4.[a,b]

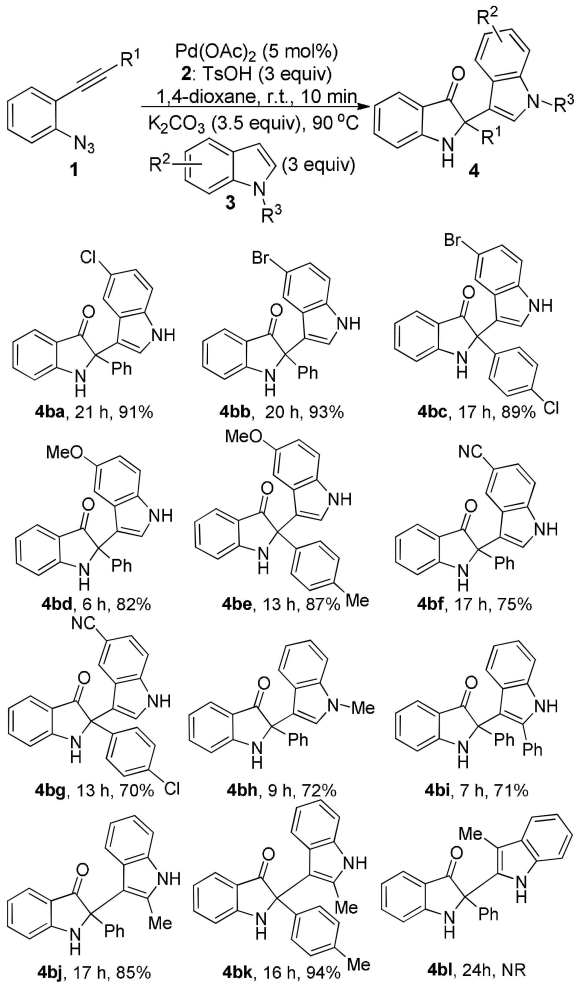


[a] Reaction conditions: **1** (0.1 mmol), $Pd(OAc)_2$ (5 mol%), **2** (3 equiv), 1,4-dioxane (1 mL), K_2CO_3 (3.5 equiv), **3a** (3 equiv), and 90 °C. [b] Isolated yields.

found to provide the desired products in moderate to excellent yields (Table 2, **4 ai-4 as**).

In contrast, reactions of 2-alkynyl arylazides with a variety of indole derivatives were also examined under the optimal reaction conditions (Table 3). Reactions of indoles bearing

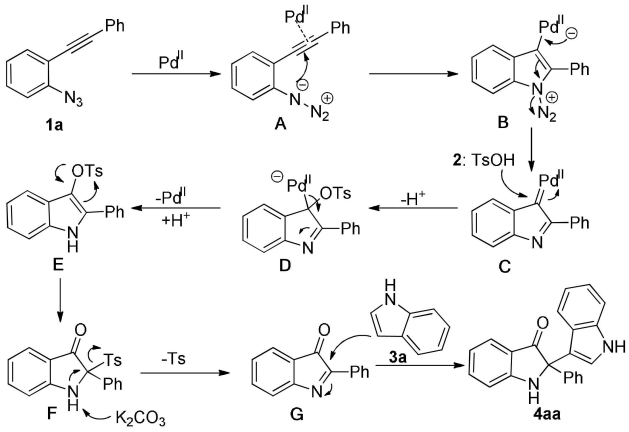
Table 3. Substrates scope to 2-(1*H*-indol-3-yl)-2-phenylindolin-3-ones.[a,b]



[a] Reaction conditions: **1** (0.1 mmol), $Pd(OAc)_2$ (5 mol%), **2** (3 equiv), 1,4-dioxane (1 mL), K_2CO_3 (3.5 equiv), **3 a** (3 equiv), and 90 °C. [b] Isolated yields.

either an electron-withdrawing or electron-donating group on the C5 carbon with alkynyl arylazides provided the desired products in 70–93% yields (Table 3, **4 ba-4 bg**). When 1-methyl-1*H*-indole was employed, the reaction was performed under the standard reaction conditions, which gave the desired product in 72% yield (Table 3, **4 bh**). Indoles with alkyl or aryl substition on the C2 carbon were also gave the crossponding products in 71–94% yields (Table 3, **4 bi-4 bk**). However, there was no desired product observed when 3-methyl-1*H*-indole was used as a nuclephile. Only the first-step intermediate 2-phenyl-1*H*-indol-3-yl 4-methylbenzenesulfonate was isolated in 83% yield (Table 3, **4 bl**).

Based on the above results, a plausible mechanism is outlined in Scheme 2, although it is highly speculative. This

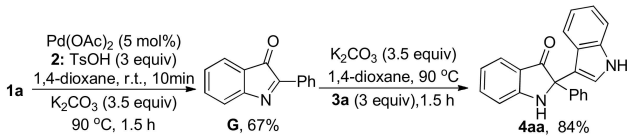


Scheme 2. Proposed mechanism.

could involve the activation of **1 a** *via* coordination of Pd catalyst to alkyne moiety and deliver the complex **A**, followed by intramolecular cyclization to give complex **B**. α-Imino palladium carbene species **C** formed by releasing the N_2, which was then trapped by TsOH to deliver the complex **D**.

Subsequent protondemetalation step gave the intermediate **E**. Under basic conditions, 2-phenyl-3*H*-indol-3-one **G** was formed *via* 1,3-Ts shit of **E** and reductive desulfonation of α-amino sulfone **F**,[9] which was further trapped by indole **3 a** to give the desired product **4 aa**.

To prove the formation of intermediate **G**, we also conducted the reaction in two steps under the optimal reaction conditions. The expected 2-phenyl-3*H*-indol-3-one **G** was isolated in 67% yield after 1.5 h, which was then trapped by indole **3 a** to give the desired product **4 aa** in 84% yield (Scheme 3).



Scheme 3. Control experiments.

Conclusions

In summary, we have successfully developed a mild and efficient synthetic method to 2-(1*H*-indol-3-yl)-2-phenylindolin-3-ones *via* $Pd(OAc)_2$-catalyzed one-pot two-step reactions of 2-alkynyl arylazides with indoles. Moreover, the reaction was shown to be applicable to various substrates, which provided the desired products up to 97% yield. Further development

and applications of this synthetic method will be disclosed in future studies.

Supporting Information Summary

Experimental details and characterization data for products (^{1}H, ^{13}C and ^{19}F NMR spectra) are available in supporting information.

Acknowledgements

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: 2-Alkynyl Arylazides · Heterocycles · 2-(1*H*-indol-3-yl)-2-phenylindolin-3-ones · One-Pot · Palladium-Catalyzed

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