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Palladium-Catalyzed Primary Amine-directed Decarboxylative Annulation of *α*-Oxocarboxylic Acids : Access to Indolo[1,2-*a*]quinazolines

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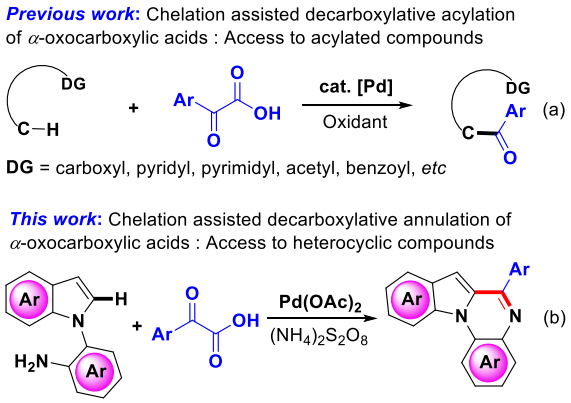
Abstract. An efficient protocol for the preparation of indolo[1,2- *a*]quinazolines via palladium-catalyzed decarboxylative annulation of indols with *α*-oxocarboxylic acids has been realized by using primary amine as a directing group (DG). This transformation proceeds smoothly with exclusive regioselectivity and represents an one-pot Domino synthesis of indo-lo[1,2- *a*]quinazolines from *α*-oxocarboxylic acids.

Keywords: palladium-catalyzed, , *α*-oxocarboxylic acids, decarboxylative annulation, directing group

Transition-metal-catalyzed decarboxylative cross-coupling is an efficient and robust synthetic protocol for carbon-carbon bond formation since it does not rely on the use of stoichiometric metallorganics and produces CO_2 instead of often toxic metal by-products.[1] Over the past decades, the decarboxylative cross-coupling reactions have achieved unprecedented development.[2] *α*-Oxocarboxylic acids have been recognized as valuable coupling reagents in the field of decarboxylation coupling reactions.[3] Therefore, to date, transition-metal-catalyzed decarboxylative cross coupling reactions involving *α*-oxocarboxylic acids have developed a widespread attention.[4] Among them, directing groups assisted decarboxylative coupling of *α*-oxocarboxylic acids are particularly attractive due to their excellent regioselectivity and reactivity.[5] For instance, Ge et al. disclosed an elegant procedure for the construction of diverse *o*-acyl acetanilides via palladium-catalyzed acylation reaction of *α*-oxocarboxylic acids and acetanilides.[6] Very recently, wang and co-workers reported that *α*-amino acids and pyridines could be used successfully as a directing group in decarboxylative coupling reaction.[7] In addition, a variety of chelating group, such as ketones,[8] indoles,[9] pyrimidines,[10] etc., have been involved in the decarboxylative coupling reactions with *α*-oxocarboxylic acids.[11] Despite such great achievements, however, in a striking contrast to the abundant protocols for the synthesis of acylated products by employing directing group strategy (Scheme 1, a), the access to functionalized heterocyclic compounds from *α*-oxocarboxylic acids is still limited (Scheme 1, b).[7a]

Scheme 1. Directing Group-Assisted Decarboxylative Cross-Coupling of *α*-Oxocarboxylic Acids



Nitrogen-containing heterocycles are important components in numerous pharmaceutical compounds and natural products compounds and natural products that exhibit a wide range of biological activities,[12] such as antitumor, anti-parasitic and antifungal. Among them, indolo[1,2- *a*]quinazolines are one of the most significant *N*-heterocycles found in functional molecules.[13] In this context, considerable efforts have been devoted to develop practical strategies for the synthesis of indolo[1,2- *a*]quinazolines.[14] Unfortunately, the efficient synthesis methods of functionalized indolo[1,2- *a*]quinazolines is rarely reported. Herein, we described a palladium catalyzed decarboxylative annulation of indoles and *α*-oxocarboxylic acids,

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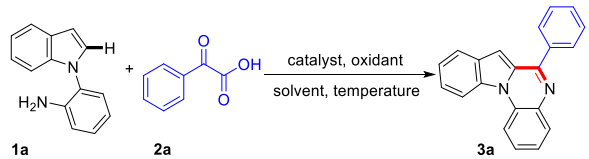
providing the indolo[1,2- *a*]quinazolines in excellent yields under mild conditions (Scheme 1, b). The transformation is probably initiated by primary amine-directed C(sp^2)-H palladation, followed by decarboxylative annulation of the *α*-oxocarboxylic acids.

Table 1. Optimization of Reaction Conditions[a]

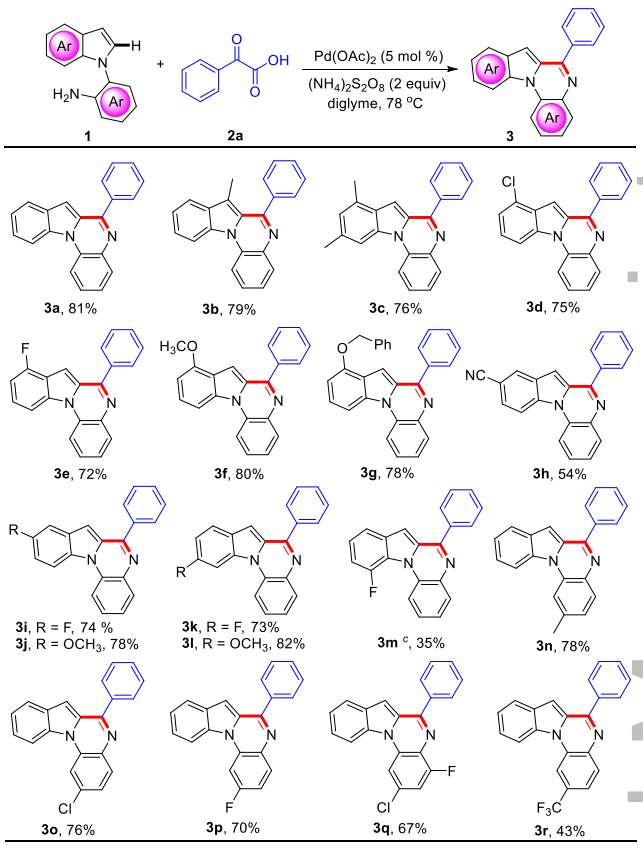


entry	catalyst	oxidant	solvent	yield[b] (%)
1	$PdCl_2$	$K_2S_2O_8$	1,4-dioxane	43
2	$Pd(OAc)_2$	$K_2S_2O_8$	1,4-dioxane	61
3	$Pd(TFA)_2$	$K_2S_2O_8$	1,4-dioxane	47
4	$Pd(PPh_3)_4$	$K_2S_2O_8$	1,4-dioxane	n.d.
5	$Pd(PPh_3)_2Cl_2$	$K_2S_2O_8$	1,4-dioxane	26
6	$Pd(OAc)_2$	$Cu(OAc)_2$	1,4-dioxane	trace
7	$Pd(OAc)_2$	AgOAc	1,4-dioxane	trace
8	$Pd(OAc)_2$	*p*-BQ[c]	1,4-dioxane	trace
9	$Pd(OAc)_2$	$(NH_4)_2S_2O_8$	1,4-dioxane	69
10	$Pd(OAc)_2$	$Na_2S_2O_8$	1,4-dioxane	52
11	$Pd(OAc)_2$	$(NH_4)_2S_2O_8$	DMSO	n.d.
12	$Pd(OAc)_2$	$(NH_4)_2S_2O_8$	DMF	n.d.
13	$Pd(OAc)_2$	$(NH_4)_2S_2O_8$	toluene	n.d.
14	**$Pd(OAc)_2$**	**$(NH_4)_2S_2O_8$**	**diglyme[d]**	**86 (81)**
15[e]	$Pd(OAc)_2$	$(NH_4)_2S_2O_8$	diglyme	75
16[f]	$Pd(OAc)_2$	$(NH_4)_2S_2O_8$	diglyme	72

[a] Reaction Conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), catalyst (5 mol %), oxidant (2 equiv) and diglyme (2.0 mL) were added to a test tube at 78 °C under air for 12 h.
[b] Determined by GC and dodecane as internal standard; Numbers in parentheses are yields of isolated; n.d. = not detected.
[c] *p*-BQ = *p*-Benzoquinone.
[d] diglyme = 1-Methoxy-2-(2-methoxyethoxy)ethane.
[e] At 70 °C.
[f] At 85 °C.

We initiated our investigation on the model reaction of 2-(1*H*-indol-1-yl)aniline (**1a**) with 2-oxo-2-phenylacetic acid (**2a**) to screen various reaction parameters. After intensive screening of the catalytic systems (Table 1), we are pleased to find the corresponding product **3a** could be obtained in 43% GC yield with $PdCl_2$ (5 mol %) as the catalyst, $K_2S_2O_8$ as the oxidant in 1,4-dioxane at 110 °C for 12 h (Table 1, entry 1). Palladium catalysts such as $Pd(OAc)_2$, $Pd(TFA)_2$, $Pd(PPh_3)_4$, and $Pd(PPh_3)_2Cl_2$ were also screened, and the results demonstrated that $Pd(OAc)_2$ was the best catalyst (Table 1, entries 2-5). Next an oxidant screening was carried out to improve the yield (Table 1, entries 6-10), and it was found that the GC yield of desired product could be improved to 69% with $(NH_4)_2S_2O_8$ as the oxidant (Table 1, entry 9). Switching the solvents to DMSO, DMF, and toluene had no better results on the reaction efficiency (Table 1, entries 11-13). To our delight, **3a** was detected in 81% isolated yield by replacing 1,4-dioxane with diglyme (Table 1, entry 10 vs 14). Finally, decreasing or increasing the reaction temperature would erode the yield of **3a** slightly (Table 1, entries 15-16).

Scheme 2. Scope of 2-(1*H*-indol-1-yl)anilines[a, b]

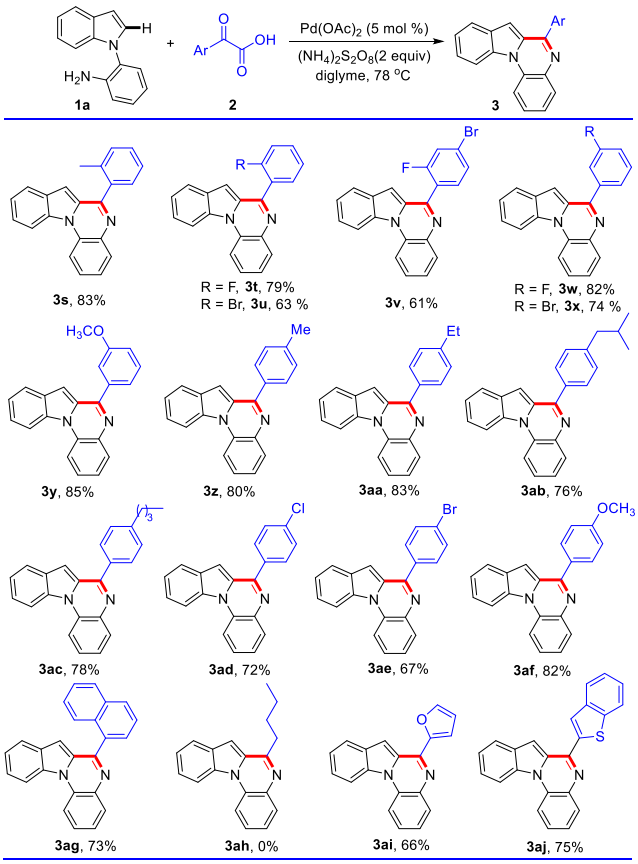


[a] Reaction conditions: a mixture of **1** (0.2 mmol), **2a** (0.4 mmol), $(NH_4)_2S_2O_8$ (0.4 mmol), $Pd(OAc)_2$ (5 mol %), and diglyme (2.0 mL) were added to a test tube at 78 °C for 12 h.
[b] Isolated yields.
[c] 4 equiv of **2m** were used.

To demonstrate the efficiency and generality of this palladium-catalyzed decarboxylative annulation reaction, we have tested the transformation of 2-oxo-2-phenylacetic acid with a variety of 2-(1*H*-indol-1-yl)anilines under the optimized reaction conditions and the representative examples are summarized in Scheme 2. Pleasingly, various 2-(1*H*-indol-1-yl)anilines with different substituent groups on the aromatic ring underwent decarboxylative annulation with 2-oxo-2-phenylacetic acid to furnish the corresponding products **3a**-**3r** in moderate to excellent yields. Substrates **1b** and **1c** with methyl-substituted gave the corresponding products **3b** and **3c** in 79% and 76% yields, respectively. The indole rings bearing either halogen (-F, -Cl), or electron-donating groups (-methoxy, -benzyloxy-), at C4, C5, C6 position were able to transfer to the desired annulation products **3d**-**3g** and **3i**-**3l** in moderate to excellent yields (72%-82%). Cyano-substituted substrate was compatible with our standard reaction

conditions, albeit in 54% yield (**3h**). Remarkably, 2-(7-fluoro-1*H*-indol-1-yl)aniline **1m** was found to yield corresponding product **3m** in an inferior yield (35%), albeit 4 equiv of **2m** were introduced incrementally. Finally, substrates with electron-donating and -withdrawing groups on the aniline rings could undergo this reaction smoothly, delivering the annulation products with good yields (**3n-3r**).

Scheme 3. Scope of *α*-oxocarboxylic acids[a, b]

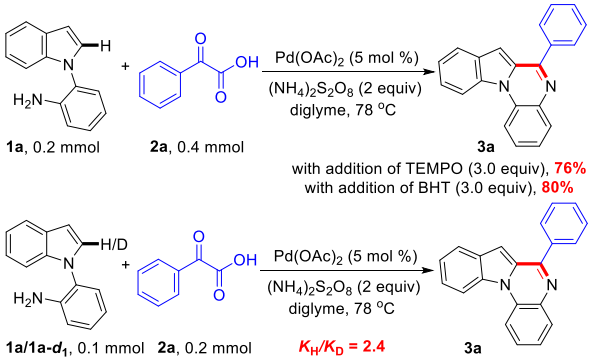


[a)] Reaction conditions: a mixture of **1a** (0.2 mmol), **2** (0.4 mmol), $(NH_4)_2S_2O_8$ (0.4 mmol), $Pd(OAc)_2$ (5 mol %), and diglyme (2.0 mL) were added to a test tube at 78 °C for 12 h

[b)] Isolated yields.

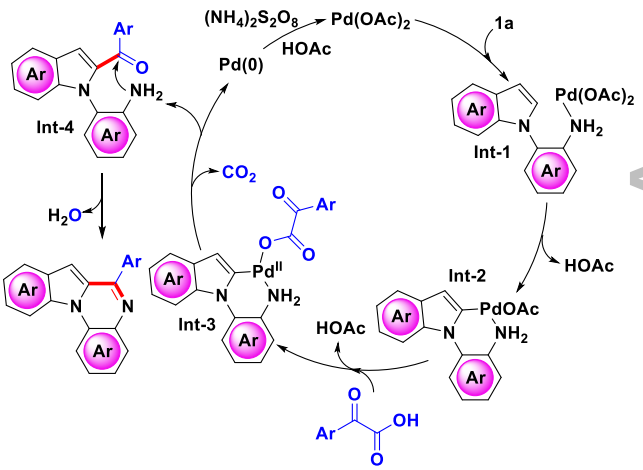
Subsequently, we examined a variety of *α*-oxocarboxylic acids and pleased to find that this decarboxylative annulation strategy was very efficient. The results were summarized in Scheme 3. Under the standard reaction conditions, a variety of 2-oxo-2-arylacetic acids with *o*-Me, *o*-F, and *o*-Br substituents were tested, delivering the expected indolo[1,2- *a*]quinazolines **3s-3u** in 63-83% yields. The annulation reaction was compatible with disubstituted 2-oxo-2-arylacetic acid to give the corresponding product in 61% yield (**3v**). To our delight, a series of *meta*-substituted 2-oxo-2-arylacetic acids, such as -F, -Br, -OCH_3 substitutions, were transformed to annulation products in acceptable yields (**3w-3y**). Similarly, the decarboxylative annulation could tolerate with either electron-donating or electron-poor groups including the CH_3, Et, *i*-Bu, *t*-Bu, OCH_3, Cl, and Br at the *para*-position, affording the corresponding products **3z-3af** in 67-83% yields. Notably, 2-(naphthalen-1-yl)-2-oxoacetic acid was also suitable for this transformation and transferred to the annulation product **3ag** in 73% yield. Unfortunately, 2-oxohexanoic acid failed to produce the annulation product under the current reaction conditions (**3ah**). Moreover, several heterocycles, related to medical chemistry,[15] including furan and benzothiophene, were also suitable substrates in this reaction conditions (**3ai-3aj**).

Scheme 4. Control Experiments



Several control experiments were subsequently conducted to develop a deeper understanding of the reaction mechanism (Scheme 4). When 3.0 equiv of radical scavengers TEMPO (2,2,6,6-tetramethyl-1-piperidinyloxy) and BHT (3,5-di-tert-butyl-4-hydroxytoluene) were added under the current reaction conditions, the corresponding annulation product **3a** was also obtained in 76% and 80% isolated yields, respectively (Scheme 4, a). This observation revealed that the reaction process should not be a radical mechanism. In addition, parallel competition reactions between 2-(1*H*-indol-1-yl)aniline (**1a**) and 2-(1*H*-indol-1-yl-2-*d*)aniline (**1a-*d*$_1$**) were carried out under the standard reaction conditions and revealed a kinetic isotope effect (k_H/k_D = 2.4, Scheme 4, b), indicating that the cleavage of C-H bond of 2-(1*H*-indol-1-yl)anilines is likely to be the rate-limiting step in the overall process.

Scheme 5. Proposed Mechanism



A plausible mechanism for this palladium-catalyzed decarboxylative annulation reaction is outlined in Scheme 5.[16][5] First, the palladacycle intermediate

Int-2 was formed via coordination of the palladium acetate to the primary amine and subsequent electrophilic attack at the C2 position of indole. Subsequently, the six-membered palladacyclic intermediate **Int 2** underwent an anion exchange with *α*-oxocarboxylic acids (**2**) to generate a cyclopalladated complex **Int-3** along with release of HOAc. Decarboxylation of **Int-3** followed by reductive elimination provides acylated compounds **Int-4**. Simultaneously, the Pd(0) can be reoxidized to the active Pd(II) species with $(NH_4)_2S_2O_8$. This acylated products then cyclized onto the carbonyl groups followed by dehydration to lead to the desired product indolo[1,2- *a*]quinazolines.

In summary, an unique and efficient decarboxylative annulation reaction of 2-(1*H*-indol-1-yl)anilines with *α*-oxocarboxylic acids has been reported via a primary amine directed C(sp^2)-H bond functionalization process. This transformation is the representative example of decarboxylative annulation of *α*-oxocarboxylic acids by employing directing group strategy. A wide range of indolo[1,2- *a*]quinazolines were obtained in moderate to excellent yield. Significantly, this protocol features exclusive regioselectivity, step economy and mild reaction conditions. We anticipate that this strategy may be applicable for other C-H functionalization/annulation reactions.

Experimental Section

Typical Experimental Procedure for Product 3

A mixture of 0.2 mmol of 2-(1*H*-indol-1-yl)anilines (**1**), 0.4 mmol of *α*-oxocarboxylic acids (**2**), 0.4 mmol $(NH_4)_2S_2O_8$, 5 mol % of $Pd(OAc)_2$, and 2.0 mL of diglyme were added to a test tube equipped with a magnetic stirring bar. The mixture was then stirred at 78 ^oC under air for 12 h. After the reaction was completed (monitored by TLC), the resulting mixture were cooled to room temperature and extracted with ethyl acetate. The combined organic layers were evaporated under vacuum. The desired products **3** were obtained in the corresponding yields after purified by column chromatography on silica gel with mixture of petroleum ether and ethyl acetate.

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Palladium-Catalyzed Primary Amine-directed Decarboxylative Annulation of α-Oxocarboxylic Acids : Access to Indolo[1,2- *a*]quinazolines

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