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Rhodium-Catalyzed Oxidative Decarbonylative Heck-type Coupling of Aromatic Aldehydes with Terminal Alkenes

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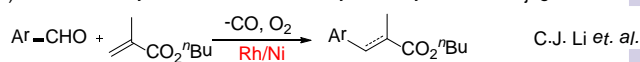
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A rhodium-catalyzed oxidative decarbonylative Heck-type coupling of aromatic aldehydes with terminal alkenes to afford 1,2-disubstituted alkenes with good regio- and E-selectivity is developed. This reaction empolys readily available aromatic aldehydes as the aryl electrophile counterpart and relies on selected acyl chloride as the crucial additive to activate the rhodium catalyst precursor.

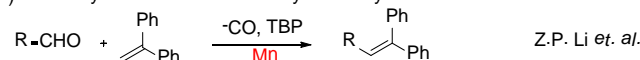
The Mizoroki-Heck reaction is a powerful and fundamental method for the direct arylation of terminal alkenes in organic synthesis.¹ Typically, these reactions employ aryl halides/sulfonates as the aryl electrophiles in the presence of suitable palladium catalysts.² Under oxidative conditions, the aryl electrophiles have been significantly expanded to include aromatic boronic acids, sulfinic acids and others.³ Meanwhile, aromatic carboxylic acids and their derivatives^{4,5} such as acyl chlorides,⁶ anhydrides⁷ and active esters⁸ can also be used in the decarbonylative Heck-type arylation of terminal alkenes. Recently, arylsulfonyl hydrazides and aroyl hydrazides have also been exemplified as potential aryl electrophiles.⁹

On the other hand, aldehydes are cheap and readily available chemicals and have been directly used as precursors for (oxidative) decarbonylative couplings catalyzed by ruthenium or rhodium, as shown by the extensive studies of C.-J. Li and co-workers since 2009.¹⁰ Among these studies, the rhodium/nickel co-catalyzed decarbonylative Heck-type coupling of aromatic aldehydes and conjugate alkenes has been established using dioxygen as oxidant, although this reaction was accompanied by the generation of conjugate addition products (Scheme 1a).^{10d} Recently, a manganese-catalyzed decarbonylative alkylation of terminal alkenes with aliphatic aldehydes has been reported by Z.-P. Li *et al.*

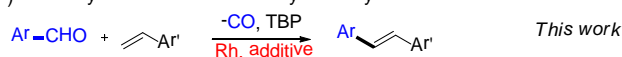
(a) Rh/Ni co-catalyzed oxidative decarbonylative arylation of conjugate alkenes



(b) Mn catalyzed oxidative decarbonylative alkylation of terminal alkene



(c) Rh catalyzed oxidative decarbonylative arylation of terminal alkene



Scheme 1 Oxidative decarbonylative couplings of aldehydes with alkenes.

(Scheme 1b).¹¹ However, in these two precedents, the former limited to conjugate alkenes as substrates, while the later limited to aliphatic aldehydes.

Besides transition-metal-catalyzed aldehydic C-H bond transformation, in the absence of transition-metal and assisted by suitable oxidants, aldehydes can also be converted into the corresponding acyl radicals for hydroacylation of olefins,¹² oxidative acylation of heterocycles¹³ and other acylative reactions,¹⁴ in which small amount of decarbonylated by-products have been detected occasionally. In our recent studies¹⁵ on the metal-free oxidative decarbonylative coupling of aromatic aldehydes with benzene, the addition of electron-deficient arenes such as *o*-dinitrobenzene as additive was critical for a moderate yield, which inspired us to use similar electron-deficient additive to approach other decarbonylative reactions. Herein, we present a general rhodium-catalyzed oxidative decarbonylative Heck-type coupling of aromatic aldehydes with terminal alkenes, and some acyl chlorides additives turned out to be crucial for the success of this reaction (Scheme 1c).

We first examined the oxidative decarbonylative coupling of *p*-cyanobenzaldehyde (**1a**) with styrene catalyzed by (CO)₂Rh(acac) using TBP (di-*tert*-butyl peroxide) as the oxidant. However, the initial attempts with electron-deficient arenes such as *o*-dinitrobenzene as additive were failed, until aroyl chloride was chosen. When 20 mol% of *p*-nitrobenzoyl chloride was used, the desired product **3a** was isolated in 58% yield (Table 1, entry 2). At the beginning, the role of aroyl chloride as the additive to this

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† Electronic Supplementary Information (ESI) available: Experimental details and characterisation of products. See DOI: 10.1039/x0xx00000x

Table 1 Optimization of the rhodium-catalyzed oxidative decarbonylative Heck-type coupling^a

entry	[Rh] (5 mol%)	Additive (mol%)	Solvent	yield [%] ^b
1	(CO) ₂ Rh(acac)	–	PhCl	< 2
2	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20)	PhCl	58
3	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (10)	PhCl	55
4	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (40)	PhCl	44
5	(CO) ₂ Rh(acac)	^p CF ₃ -C ₆ H ₄ COCl (20)	PhCl	48
6 ^c	(CO) ₂ Rh(acac)	(NO ₂) ₂ -C ₆ H ₃ COCl (20)	PhCl	46
7	(CO) ₂ Rh(acac)	PhCOCl (20)	PhCl	51
8	(CO) ₂ Rh(acac)	^t BuCOCl (20)	PhCl	39
9	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20) K ₂ CO ₃ (20)	PhCl	50
10	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20) Et ₃ N (20)	PhCl	56
11	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20) Ph ₃ P (20)	PhCl	0
12	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20) 1,10-phen (20)	PhCl	0
13	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20)	DCE	48
14	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20)	toluene	56
15	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20)	dioxane	14
16 ^d	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20)	PhCl	44
17 ^e	(CO) ₂ Rh(acac)	^p NO ₂ -C ₆ H ₄ COCl (20)	PhCl	73
18 ^e	[(CO) ₂ RhCl] ₂	^p NO ₂ -C ₆ H ₄ COCl (20)	PhCl	63
19 ^e	[(COD)RhCl] ₂	^p NO ₂ -C ₆ H ₄ COCl (20)	PhCl	82
20 ^e	[(COD)RhCl] ₂	PhCOCl (20)	PhCl	83

^a Conditions: **1a** (0.2 mmol), styrene (4 equiv, 0.8 mmol), rhodium catalyst, additives, TBP (2.5 equiv, 0.5 mmol), chlorobenzene (0.5 mL), reacted for 12 h at 140 °C under argon atmosphere unless otherwise noted. ^b Isolated yields. ^c 3,5-Dinitrobenzoyl chloride was used. ^d Reacted at 120 °C. ^e Catalyst loading increased to 10 mol% (based on Rh).

reaction was unclear, so the dosage of *p*-nitrobenzoyl chloride and other different aryl chlorides including *p*-trifluoromethylbenzoyl chloride, 3,5-dinitrobenzoyl chloride, benzoyl chloride and pivaloyl chloride were tried, which all resulted in lower yields (entries 3–8). By isolating and characterizing the reaction mixture carefully, benzoyl chloride was proved to react with the excess styrene and convert to stilbene, which was similar to the report by Miura *et al.* (for details, see Scheme 2).^{6c, 6d} So inorganic and organic bases were added to neutralize the hydrochloride released from the benzoyl chloride, and slightly lower yields were obtained (entries 9 and 10). Next, phosphine- and nitrogen- containing ligands were tried, leading to total deactivation of the catalysts, maybe due to coordination saturation of rhodium (entries 11 and 12). This reaction can also occur in dichloroethane (DCE) or toluene, and similar yields were obtained; while in dioxane, the yield decreased drastically (entries 13–15). Reacting at 140 °C was necessary for the

total conversion of *p*-cyanobenzaldehyde (**1a**), and lower yield was obtained when reacted at 120 °C (entry 16). To our delight, a better

Table 2 The influence of aromatic aldehydes on the oxidative decarbonylative Heck-type coupling^a

Ar-CHO	Yield [%]	E/Z Ratio
1a-1o		
3a , 83%		
3b , 73%		
3c , 61%		
3d , 55%		
3e , 51%		
3f , 53%		
3g , 46% ^b		
3h , 76%, E/Z = 94:6		
3i , 61%, E/Z = 85:15		
3j , 59%		
3k , 65%		
3l , 67%, E/Z = 75:25		
3m , 66%		
3n , 58%		

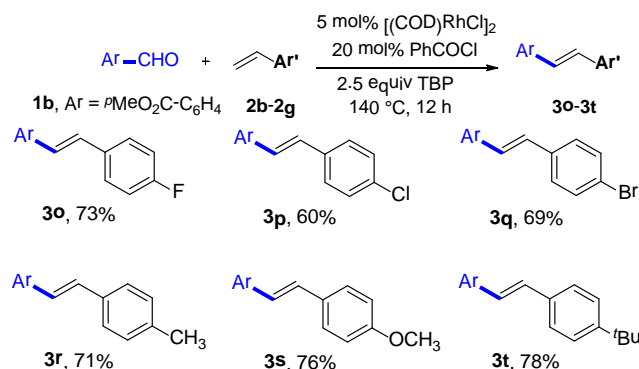
^a Conditions: **1** (0.2 mmol), styrene (4 equiv, 0.8 mmol), [(COD)RhCl]₂ (5 mol%, 0.01 mmol), PhCOCl (20 mol%, 0.04 mmol), TBP (2.5 equiv, 0.5 mmol), chlorobenzene (0.5 mL), reacted for 12 h at 140 °C under argon atmosphere unless otherwise noted. Isolated yields were reported. The E/Z ratio > 20:1 (determined by crude ¹H NMR) unless otherwise noted. ^b *p*-Nitrobenzoyl chloride was used instead of benzoyl chloride.

yield was realized by increasing the catalyst loading to 10 mol% (entry 17). By comparing several common used Rh(I) catalysts, [(COD)RhCl]₂ revealed as the best choice, leading to a yield of 82% (entry 19). Similarly, with 10 mol% [(COD)RhCl]₂ as the catalyst benzoyl chloride as additive afforded a slightly higher yield (83%, entry 20). It's worth noting that the *trans* isomer was obtained predominately (*trans*:*cis* > 98:2) under the optimized conditions as determined by ¹H NMR of the crude reaction mixture, and 1,1-diarylethene was not detected.

The generality of this oxidative decarbonylative coupling of aromatic aldehydes with terminal alkenes was subsequently investigated with benzoyl chloride as the additive. The effect of substituents on the aromatic aldehyde moiety is listed in Table 2. Benzaldehydes bearing electron withdrawing or donating substituents were successfully transformed into the desired decarbonylated Heck-type coupling products in moderate yields, such as cyano (**1a** and **1h**), methoxycarbonyl (**1b** and **1j**), trifluoromethyl (**1c**), halo (**1d-1f**, **1i** and **1j**), and methoxyl group (**1k** and **1m**). Besides substituted benzaldehydes, the optimized reaction conditions can also be applied to the decarbonylative coupling of β-naphthaldehyde (**1n**) with styrene. To our delight, the substitution position (*para*, *meta* or *ortho*) did not show much influence on the yields, albeit the E/Z ratios dropped for *ortho*-substituted **3l**. However, this oxidative decarbonylative coupling failed for the aliphatic aldehydes such as *n*-butanal; 0

corresponding coupling product was detected by the GC-MS and the aliphatic carboxylic acid was revealed as the main product.

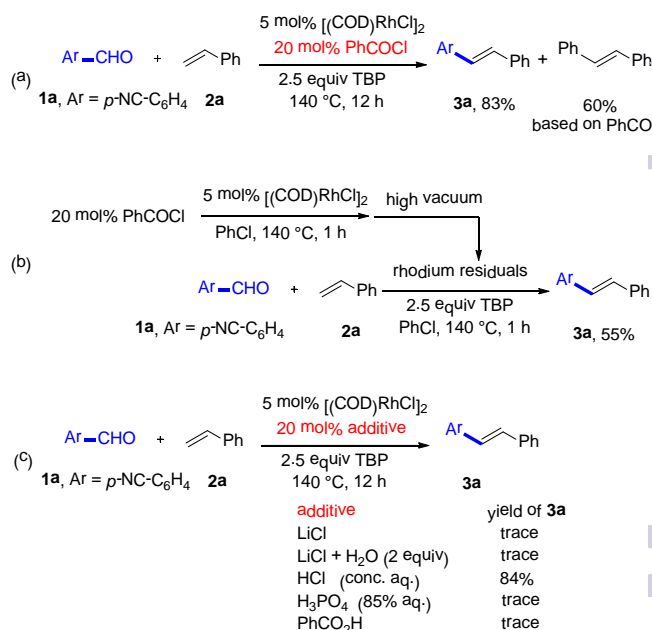
Table 3 The influence of terminal alkenes on the oxidative decarbonylative Heck-type coupling^a



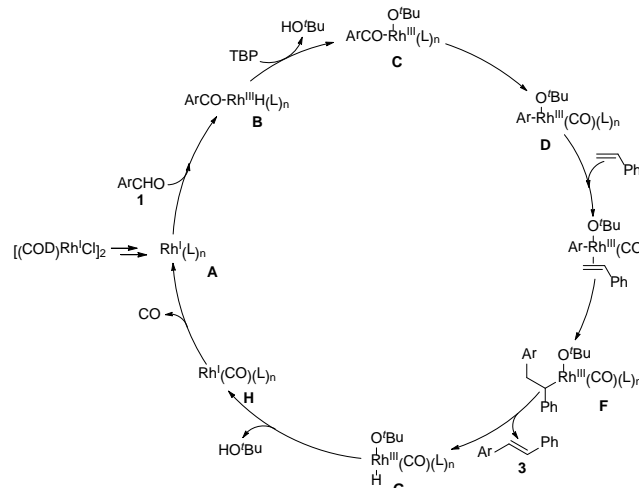
^a Conditions: **1a** (0.2 mmol), **2** (4 equiv, 0.8 mmol), [(COD)RhCl]₂ (5 mol%, 0.01 mmol), PhCOCl (20 mol%, 0.04 mmol), TBP (2.5 equiv, 0.5 mmol), chlorobenzene (0.5 mL), reacted for 12 h at 140 °C under argon atmosphere unless otherwise noted. Isolated yields were reported. The E/Z ratio > 20:1 (determined by crude ¹H NMR).

After investigating the scope of aromatic aldehydes, we next tested the generality of this oxidative decarbonylative coupling of *p*-methoxycarbonyl benzaldehyde (**1b**) with substituted styrenes (**2b-2g**),¹⁶ affording structurally diverse 1,2-diaryl alkenes in moderate to good yields with excellent regio- and *trans*-selectivity.

The selected acyl chloride played a critical part in this rhodium-catalyzed oxidative decarbonylative Heck-type coupling, however, its specific role was unclear. So, some mechanistic experiments were designed and performed to shed some light on the mechanism. Firstly, by carefully isolating and characterizing of the reaction mixture, benzoyl chloride was proved to react with the excess styrene and convert to stilbene in 60% yield (based on benzoyl chloride, Scheme 2a), which was similar to the report by Miura *et al.*^{6d} Considering the same reaction without adding benzoyl chloride resulted in an extremely low yield (< 2%, Table 1, entry 1), and benzoyl chloride was very reactive and would react with [(COD)RhCl]₂ much faster than *p*-cyanobenzaldehyde (**1a**), it is very probably that the rhodium catalyst precursor was activated in the catalytic cycle for the generation of stilbene. This assumption was strongly supported by the stepwise reaction (Scheme 2b): the rhodium catalyst precursor reacted with benzoyl chloride in the absence of styrene for 1 h at 140 °C, and then evaporated under high vacuum (under 1 mmHg for 12 h) to afford rhodium residuals, which relayed to catalyze the oxidative decarbonylative Heck-type coupling of *p*-cyanobenzaldehyde (**1a**) and styrene to produce **3a** in 55% yield (Scheme 2b). Secondly, control experiments using LiCl, H₃PO₄ and benzoic acid (20 mol%) to replace benzoyl chloride failed in the model reaction (Scheme 2c). However, when aqueous HCl was tested similarly, the reaction proceeded smoothly with almost the same yield as benzoyl chloride (the influence of water was also eliminated). These results revealed that the chloride anion or the proton alone was ineffective but hydrochloride was effective additive, which implied hydrochloride was able to activate the rhodium catalyst precursor, most probably via oxidative addition with the catalyst precursor as the benzoyl chloride.



Scheme 2 Mechanistic experiments to understand the role of acyl chloride.



Scheme 3 Proposed mechanism for this oxidative decarbonylative coupling of aldehydes with alkenes.

Based on the literature reports and our studies, a plausible mechanism was proposed in Scheme 3. First, catalyst precursor [(COD)RhCl]₂ undergoes oxidative addition with benzoyl chloride, decarbonylation, insertion into C=C bond of styrene, β-hydride elimination and reductive elimination to generate stilbene and the activated rhodium catalyst (A, abbreviated to Rh^I(L)_n), according to the route reported by Miura *et al.*^{6d} Then, oxidative addition of aldehydic C-H bond with the activated rhodium catalyst takes place to afford aroyl rhodium hydride B. The hydride was oxidized by TBP, further decarbonylated to give rhodium complex D. Next, the coordination of styrene to rhodium complex D, C=C bond insertion, β-hydride elimination provides the oxidative decarbonylative Heck-type coupling product (**3a**) and rhodium complex G; further reductive elimination and dissociation of carbon monoxide from rhodium re-generates the activate rhodium catalyst (A).

Conclusions

We have developed an efficient rhodium-catalyzed oxidative decarbonylative coupling of aromatic aldehydes with terminal alkenes to produce 1,2-biaryl alkenes. Some acyl chlorides have been proved as critical additives for the success of this reaction, and most probably activate the rhodium catalyst precursor by undergoing decarbonylative coupling with styrene. This method not only broadened the substrate scope for oxidative decarbonylative coupling of aldehydes with alkenes based on previous reports, but also offers a new strategy for the *in situ* activation of rhodium catalysts.

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The authors declare no competing financial interest.

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- The decarbonylative Heck-type coupling of aromatic aldehyde and alkyl substituted alkenes such as 1-octene afforded a mixture of region-isomers due to the C=C bond immigration.

Abstract for the Contents Pages

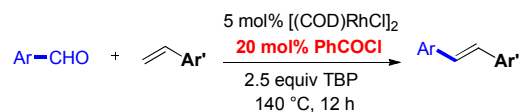
Rhodium-Catalyzed Oxidative Decarbonylative Heck-type Coupling of Aromatic Aldehydes with Terminal Alkenes

Lei Kang, Feng Zhang, Lin-Ting Ding and Luo Yang*

Text:

A rhodium-catalyzed oxidative decarbonylative Heck-type coupling of aromatic aldehydes and terminal alkenes to afford 1,2-disubstituted alkenes with good regio- and E-selectivity is developed.

Graphic:



Advantages:

- readily available aldehydes as aryl electrophile
- acyl chloride as crucial additive to activate the catalyst precursor
- good regio- and E-selectivity