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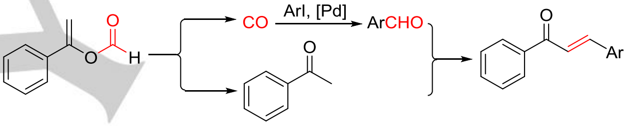
1-Arylvinyl formats: A New CO Source and Ketone Source in Carbonylative Synthesis of Chalcone Derivatives

Xinxin Qi,[a] Ming Lai,[a] Min-Jie Zhu,[a] Jin-Bao Peng,[a] Jun Ying,[a] Xiao-Feng Wu*[a,b]

Abstract: 1-Arylvinyl formates as a kind of new CO surrogate have been explored for the first time. Most of the known CO precursors usually produce undesired residuals, which have to be removed. In this strategy, after CO release, the in situ generated acetophenones from 1-arylvinyl formates can be successfully applied as a good ketone source in the synthesis of chalcones with benzaldehydes *via* a palladium-catalyzed reductive carbonylation reaction. A variety of chalcones were isolated in satisfactory to good yields with good substrates compatibilities under mild conditions.

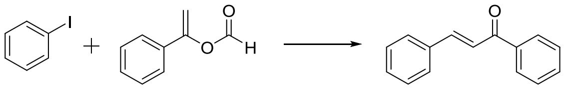
Since the pioneering work of Heck and co-workers in 1974,[1] palladium-catalyzed carbonylative transformations have drawn continuously attentions for their widely application in the synthesis of numerous congeners of carbonyl compounds.[2] As a fundamental C1 source, carbon monoxide (CO) gas is cheap and easily available, it has been widely utilized in palladium-catalyzed carbonylative transformations, especially in industrial scales. However, gaseous CO is odorless, toxic, and requires high pressure-resistant equipment. All of these characters of CO gas restrict its utilization on a laboratory scale. Therefore, many efforts have been put in alternative methods with CO gas-free conditions, and various CO surrogates have been development in recent years, such as metal carbonyl complexes,[3] oxalyl chlorides,[4] paraformaldehyde,[5] formates,[6] formamides,[7] benzene-1,3,5-triyl triformate (TFBen),[8] and others.[9] However, the known CO precursors have several disadvantages, including harsh reaction conditions, (sub)stoichiometric metal wastes or other unneeded residuals, and so on. In this sense, the searching for new CO sources without byproducts affecting under mild reaction conditions are still highly in demand.

Chalcones and their derivatives represent a valuable class of natural products, which display a wide spectrum of biological activities.[10] They also serve as very important synthons for heterocycle preparation. Due to their versatile bioactive properties and promising application for the investigation of new drugs, research on chalcones and their derivatives have drawn continuously attention.[11] Traditional synthetic methods for chalcones mainly rely on Claisen-Schmidt condensation reaction of aromatic aldehydes with acetophenones under strong basic conditions.[12] And in recent years, palladium-catalyzed carbonylative heck-type coupling reaction between aryl triflates/halides with diverse vinyl-containing compounds under CO pressure have become a powerful strategy for the synthesis of chalcones and their analogs.[13] In our continuous efforts on the development of new CO surrogates, we recently reported a series of palladium-catalyzed carbonylation reaction with formic acid as the CO source.[14] However, with acetic anhydrides as activator, the in situ generated acetic acid need excess amount of base to neutralize. Considering both the synthetic interest for chalcones and the investigation of new CO precursors, compound 1-phenylvinyl formate came into our mind. It would release CO and acetophenone under base condition. The in situ formed CO could react with iodobenzene *via* a reductive carbonylation reaction to give benzaldehyde, followed by an aldol condensation with residual acetophenone to afford chalcone product (**Scheme 1**). With this idea in mind, we describe here a palladium-catalyzed reductive carbonylation reaction for the synthesis of chalcones and their derivatives from aryl iodides with 1-arylvinyl formates as both CO source and ketone source.



Scheme 1. Proposed method for carbonylative synthesis of chalcone with 1-phenylvinyl formate as the new designed CO surrogate.

Table 1. Screening of reaction conditions.^{a}



1a **2a** **3aa**

Entry	Ligand	Base	Solvent	Yield (%)^{b}
1	PCy_3	DBU	1,4-dioxane	33
2	PPh_3	DBU	1,4-dioxane	22
3	$BuPAd_2$	DBU	1,4-dioxane	23
4	**L1**	DBU	1,4-dioxane	61
5	Xantphos	DBU	1,4-dioxane	36
6	DPPB	DBU	1,4-dioxane	17
7	DPPPE	DBU	1,4-dioxane	19
8	DPPF	DBU	1,4-dioxane	16
9	**L1**	Et_3N	1,4-dioxane	0
10	**L1**	DiPEA	1,4-dioxane	0
11	**L1**	DABCO	1,4-dioxane	0
12	**L1**	NaHMDS	1,4-dioxane	0
13	**L1**	Cs_2CO_3	1,4-dioxane	0
14	**L1**	KOH	1,4-dioxane	0
15	**L1**	DBU	THF	43

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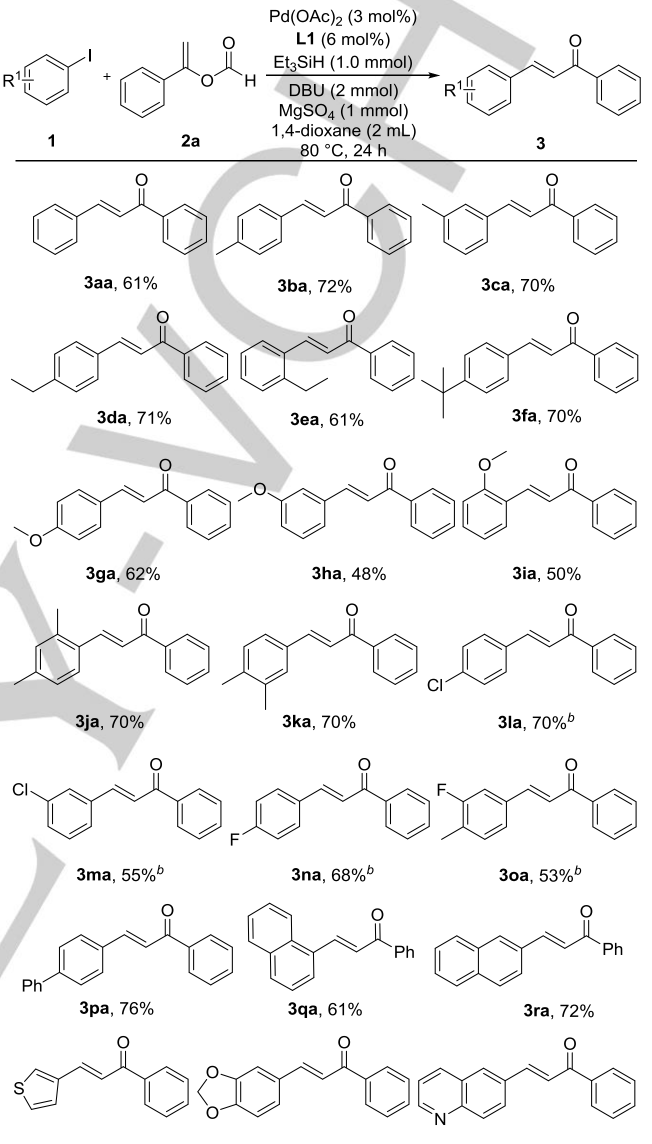
16	**L1**	DBU	CH_3CN	49
17	**L1**	DBU	DMF	32
18	**L1**	DBU	DMSO	18
19	**L1**	DBU	Toluene	45
20[c]	**L1**	DBU	1,4-dioxane	65

[a] Reaction conditions: iodobenzene (0.5 mmol), 1-phenylvinyl formate (1.5 mmol), catalyst (3 mol%), ligand (3 or 6 mol%), base (2.0 mmol), Et_3SiH (1.0 mmol), solvent (2 mL), 80 °C, 24 h. [b] Yield were determined by GC with dodecane as an internal standard. [c] $MgSO_4$ (1.0 mmol). L1: 2-(diphenylphosphino)biphenyl.

Initially, iodobenzene was used as model substrate, $Pd(OAc)_2$ as catalyst, PCy_3 as ligand, Et_3SiH as hydrogen source, DBU as base, 1-phenylvinyl formats as both CO and ketone source, in 1,4-dioxane at 80°C. Fortunately, 33% yield of the desired product was obtained (Table 1, entry 1). Encouraged by this result, various mono- and bidentate phosphine ligands were studied, involving PPh_3, $BuPAu_2$. **L1** [2-(diphenylphosphino)biphenyl], Xantphos, DPPB, DPPPE, and DPPF (Table 1, entries 2-8). 61% yield was obtained with **L1** as the ligand (Table 1, entry 4). It was noteworthy that the base played a very important role in this reaction, no product could be observed with Et_3N, DiPEA, DABCO, NaHMDS, Cs_2CO_3, or KOH as the base (Table 1, entries 9-14). Furthermore, screening of different solvents proven 1,4-dioxane to be the best solvent for the transformation (Table 1, entries 15-19). Remarkably, a slightly higher yield can be achieved when $MgSO_4$ was used as an additive (Table1, entry 20). Here, the role of $MgSO_4$ is to absorb the water generated from aldol condensation between acetophenone and benzaldehyde.

With the optimized reaction conditions in hands, we went on our study on substrates scope and the results were summarized in Table 2. Aryl iodides with electron-donating groups, involving methyl, ethyl, *t*-butyl, methoxy and dimethyl-substitute groups are tolerated well to give the corresponding chalcone products in good yields (**3aa-3ka**). Notably, those substrates bearing *ortho-* and *para*-substitute afforded the target products in higher yields compared with *meta*-substituted starting materials, which might due to the electronic effect (**3ga, and 3ia** vs. **3ha**). Aryl iodides with chloro and fluoro groups could smoothly produce the final products in 55% and 68% yields as well (**3la**-**3an**). Notably, aryl iodides bearing both methyl and fluoro groups were also worked well to provide the corresponding chalcone in moderate yield (**3oa**). Furthermore, substrates with 1-biphenyl, 1- and 2-naphthalene moieties were examined; the desired chalcone products were obtained in good yields (**3pa-3ra**). We also tested heterocycles such as 3-thiophene, 5-benzodioxole, and 6-iodoquinoline groups; the desired products were isolated in 50%, 52%, and 58% yields, respectively (**3sa-3ua**).

Table 2. Substrates scope on aryl iodides.[a]



3aa, 61% **3ba**, 72% **3ca**, 70%

3da, 71% **3ea**, 61% **3fa**, 70%

3ga, 62% **3ha**, 48% **3ia**, 50%

3ja, 70% **3ka**, 70% **3la**, 70%[b]

3ma, 55%[b] **3na**, 68%[b] **3oa**, 53%[b]

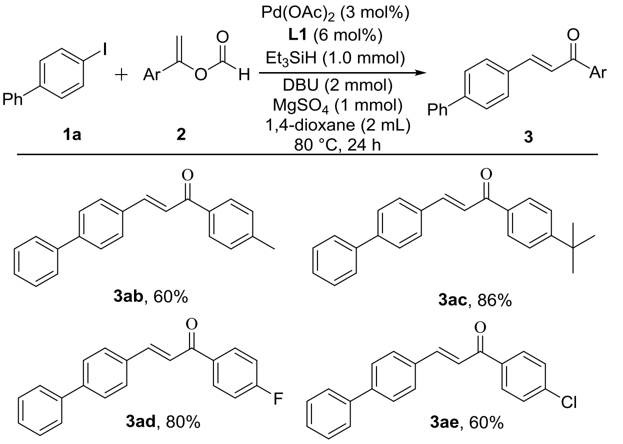
3pa, 76% **3qa**, 61% **3ra**, 72%

3sa, 50% **3ta**, 52% **3ua**, 61%[b]

[a] Reaction conditions: aryl iodides (0.5 mmol), 1-phenylvinyl formate (1.5 mmol), $Pd(OAc)_2$ (3 mol%), **L1** (6 mol%), DBU (2.0 mmol), Et_3SiH (1.0 mmol), $MgSO_4$ (1.0 mmol), 1,4-dioxane (2 mL), 80 °C, 24 h, isolated yield. [b] 60 °C.

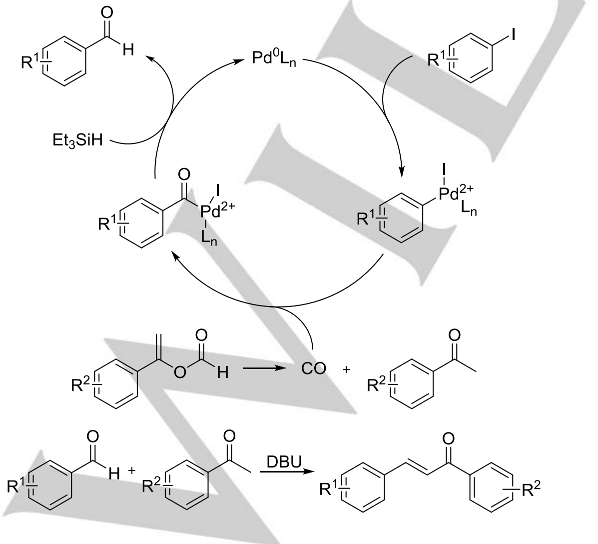
Furthermore, the substrates scope of 1-arylvinyl formats have been investigated (Table 3). 1-Arylvinyl formats with methyl, *t*-butyl, fluoro, and chloro groups worked well to produce the chalcone derivatives in moderate to good yields (**3ab**-**3ae**).

Table 3. Substrate scopes on 1-arylvinyl formats.[a]



[a]Reaction conditions: iodobenzene (0.5 mmol), 1-arylvinyl formates (1.5 mmol), $Pd(OAc)_2$ (3 mol%), **L1** (6 mol%), DBU (2.0 mmol), Et_3SiH (1.0 mmol), $MgSO_4$ (1.0 mmol), 1,4-dioxane (2 mL), 80 °C, 24 h, isolated yield.

Based on the above results, a plausible reaction mechanism is proposed (**Scheme 2**). First, arylpalladium complex was formed from the oxidative addition of Pd^0L_n with aryl iodide. Subsequently, benzoylpalladium intermediate will be formed after the coordination and insertion of CO (in situ generated from 1-arylvinyl formates) to the arylpalladium complex, which will finally eliminate aromatic aldehydes in the presence of Et_3SiH. Furthermore, an aldol condensation between produced aromatic aldehydes and acetophenones (in situ generated from 1-arylvinyl formates) in the presence of DBU, affording the final chalcone products.



Scheme 2. Proposed reaction mechanism.

In conclusion, 1-arylvinyl formats as a kind of new CO precursors have been prepared and applied. As both promising CO source and ketone source, 1-arylvinyl formates have been successfully utilized in the synthesis of chalcone derivatives through a palladium-catalyzed reductive carbonylation reaction with aryl iodides for the first time. A series of aryl iodides were tolerated well to provide the final chalcone products in moderate to high yields under mild reaction conditions. The further applications of this kind of CO surrogate are under progress.

Experimental Section

Under nitrogen, $Pd(OAc)_2$ (3 mol%), **L1** ([1,1'-biphenyl]-2-yldiphenyl phosphine) (6 mol%) and $MgSO_4$ (1.0 mmol) were added to a 15 mL tube. Then iodobenzene (0.5 mmol), 1-arylvinyl formate (1.5 mmol), Et_3SiH (1.0 mmol) and DBU (2.0 mmol) were added by syringe. The tube was sealed and the reaction mixture was stirred at 60-80 ^oC for 24 h. After the reaction was completed, the reaction mixture was concentrated by rotary evaporation. The crude mixture was purified by silica gel column chromatography (PE/EA = 50/1) to provide the desired pure products.

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Keywords: CO surrogate • 1-arylvinyl formates • chalcones • palladium catalyst • reductive carbonylation

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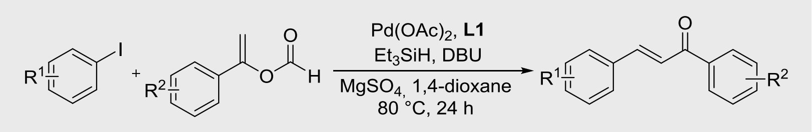

1-Arylvinyl formates as a kind of efficient and convenient CO surrogates have been explored for the first time. In this strategy, after CO release, the in situ generated acetophenones from 1-arylvinyl formates could be successfully applied as a good ketone source in the synthesis of chalcones with benzaldehydes *via* a palladium-catalyzed reductive carbonylation reaction. A variety of chalcones were isolated in moderate to excellent yields with good substrates compatibilities under mild

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