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To be cited as: *Chem. Eur. J.* 10.1002/chem.201604256

Link to VoR: http://dx.doi.org/10.1002/chem.201604256

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DOI: 10.1002/chem.201xxxxxx
CHEMISTRY
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CO_2 Fixation

Palladium-Catalyzed Multi-Component Reactions of *N*-Tosylhydrazones, 2-Iodoanilines and CO_2 towards 4-Aryl-2-Quinolinones

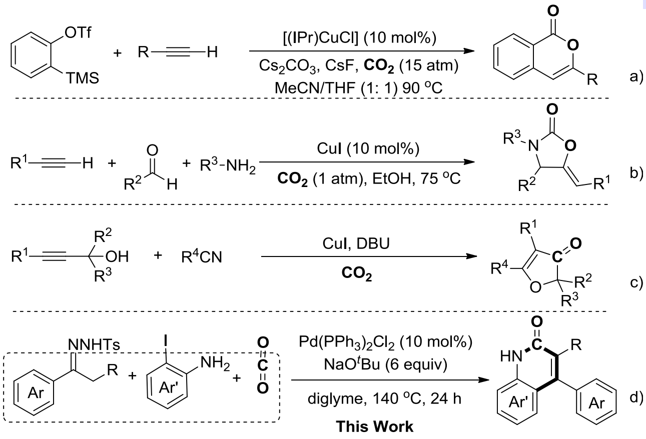
Song Sun, Wei-Ming Hu, Ning Gu, Jiang Cheng*

Dedication ((optional)

Abstract: A palladium-catalyzed three-component reaction between *N*-tosylhydrazones, 2-iodoanilines and atmospheric pressure of CO_2 was developed whereby a tandem carbene migration insertion/lactamization strategy to afford 4-aryl-2-quinolinones in moderate to good yields. Notablely, a wide range of functional groups were tolerated in this procedure. This protocol features with simultaneous formation of four novel bonds i.e. two C–C, one C=C and one C-N bonds in amide, representing an efficient methodology for incorporation of CO_2 into heterocycles.

Recently, the incorporation of the entire "CO_2" moiety or its "C=O" fragment via multi-component reactions towards carbonyl-containing compounds,[1-2] especially heterocycles,[3] represents a great advance and greener approach by avoiding the use of hazardous carbonylation reagents such as carbon monoxide and phosgene. With this regard, Kobayashi, Kunai, as well as others independently reported elegant cascade reaction of *in situ* formed benzyne with a nucleophile and CO_2 towards a series of carbonyl-containing heterocycles (Scheme 1a).[4] The sequential carboxylation/cyclization reaction of propargylic alcohols (or amines), CO_2 and a third component allows to facilely construction of oxazolidinones or other useful heterocyclic frameworks.[5] For instance, Li developed a four-component reaction of aldehydes, primary amines, alkynes, and CO_2 leading to oxazolidinones (Scheme 1b).[6] Jiang reported copper-catalyzed domino reaction of readily available propargylic alcohols, CO_2 and nitriles to access highly substituted 3(2*H*)-furanones (Scheme 1c).[7] However, despite much progress has been made in this field, the development of procedures involving either new reaction partners or new target molecules in the fixation of CO_2 remains an area of synthetic interest.

On the other hand, thanks to the well-developed *N*-tosylhydrazone chemistry,[8-9] the reaction of *N*-tosylhydrazone with aryl- halides generates corresponding olefins whereb sequential carbene migration insertion/β-H eliminatio procedure.[10] Therefore, in the case of 2-iodoaniline, *ortho*-(1 phenylvinyl)aniline is formed,[10c] which is a potential substrate t incorporate CO_2 towards 2-quinolinones developed by Yu[11a] an others recently.[11] Thus, a three-component reaction between N tosylhydrazones, 2-iodoanilines and CO_2 was highly expected i the construction of 2-quinolinones, which did not require the time consuming procedure for the isolation of the intermediates *orthc* (1-phenylvinyl)anilines. As part of our continuing studies on th transformation of CO_2,[12] we herein present an unprecedente strategy towards 4-aryl-2-quinolinones by a three-componer coupling reaction of *N*-tosylhydrazones, 2-iodoanilines and CO catalyzed by a Pd(II)/phosphine system (Scheme 1d). Suc frameworks were ubiquitous in drugs, bioactive natural products photoelectric materials, and important intermediates in organi synthesis.[13]



Scheme 1. Some typical examples of multi-component reaction involving carbon dioxide towards carbonyl-containing heterocycles.

Initially, we tested the reaction of *N*-tosylhydrazone **1a** (0.3 mmol), 2-iodoaniline **2a** (0.2 mmol), and atmospheric pressure of CO_2 (Table 1). To our delight, this three-component reaction took place in the presence of $Pd(OAc)_2$ (10 mol%) and NaO^tBu (1.2 mmol) under atmospheric pressure of CO_2 in diglyme at 140 °C for 24 h, producing 4-phenyl-2-quinolinone **3a** in 27% yield (Table 1, entry 1). The employment of phosphine ligands increased the reaction efficiency dramatically, and PPh_3 was the best (65%, Table 1, entries 2-4) (for details, see Supporting Information). The efficiency of the reaction was further increased to 68% by

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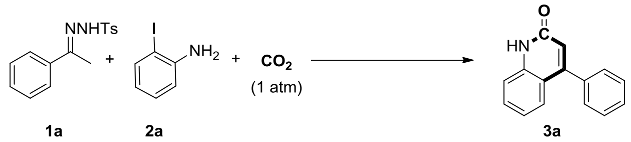
[*] [a] Dr. S. Sun, W.-M. Hu, N. Gu, and Prof. Dr. J. Cheng
School of Petrochemical Engineering, Jiangsu Key Laboratory of Advanced Catalytic Materials & Technology, and Jiangsu Province Key Laboratory of Fine Petrochemical Engineering, Changzhou University, Gehu Road 1, Changzhou, 213164, P. R. China
E-mail: jiangcheng@cczu.edu.cn
Homepage: www.chengjiangjs.com
Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/chem.201xxxxxx.

replacing $Pd(OAc)_2/PPh_3$ with $Pd(PPh_3)_2Cl_2$ (Table 1, entries 5-7). Among the bases tested, NaO^tBu was the best (68%, Table 1, entries 7-9). The solvent had a profound effect on the reaction and diglyme (68%) was superior to 1, 4-dioxane (53%), while DMSO almost inhibited the reaction (Table 1, entries 7, 10-11). Notablely, in the presence of 6 equivalents of NaO^tBu, **3a** was isolated in 75% yield (**1a**: **2a** = 1.7: 1, Table 1, entry 12). Increasing the pressure of CO_2 did not have any positive effect for this transformation (Table 1, entry 13).

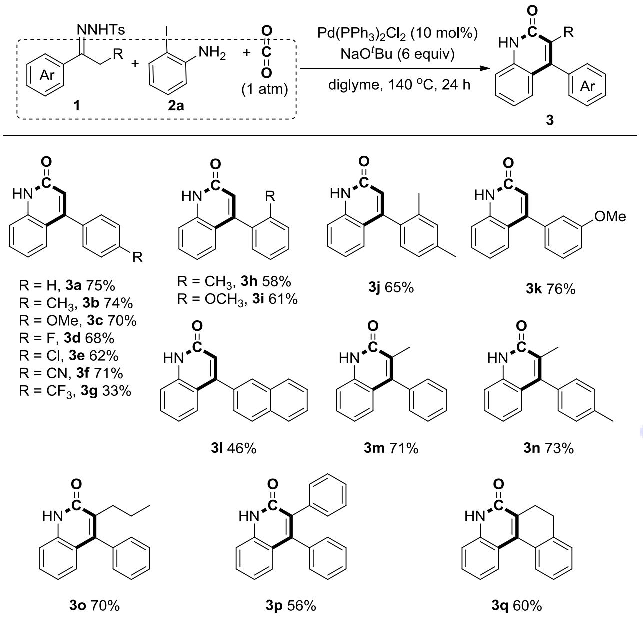
Table 1. Selected results for screening the optimized reaction conditions[a]



Entry	[Pd]	Ligand	Base	Isolated Yield [%]
1	$Pd(OAc)_2$	--	NaO^tBu	27
2	$Pd(OAc)_2$	Xphos	NaO^tBu	51
3	$Pd(OAc)_2$	Ph_2P(2-Py)	NaO^tBu	62
4	$Pd(OAc)_2$	PPh_3	NaO^tBu	65
5	$PdCl_2$	PPh_3	NaO^tBu	64
6	$Pd(PPh_3)_4$	--	NaO^tBu	45
7	$Pd(PPh_3)_2Cl_2$	--	NaO^tBu	68
8	$Pd(PPh_3)_2Cl_2$	--	LiO^tBu	48
9	$Pd(PPh_3)_2Cl_2$	--	KO^tBu	< 5
10[b]	$Pd(PPh_3)_2Cl_2$	--	NaO^tBu	53
11[c]	$Pd(PPh_3)_2Cl_2$	--	NaO^tBu	0
12	$Pd(PPh_3)_2Cl_2$	--	NaO^tBu	51[d] 71[e] 75[f] 72[g]
13[h]	$Pd(PPh_3)_2Cl_2$	--	NaO^tBu	41

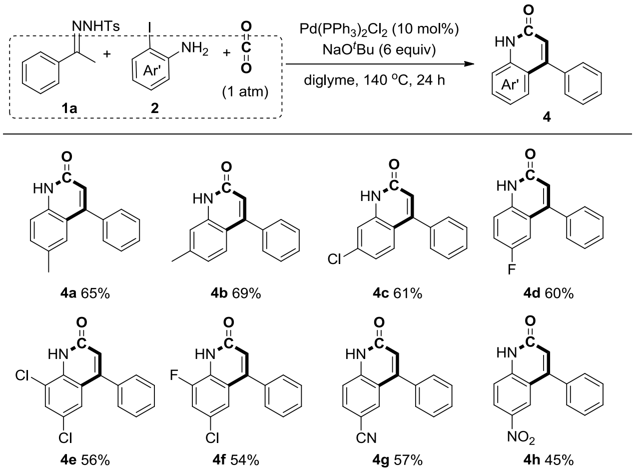
[a] Reaction conditions: **1a** (0.3 mmol), **2a** (0.2 mmol), [Pd] (0.02 mmol), ligand (0.04 mmol), base (1.2 mmol), diglyme (3.0 mL), at 140 °C under atmospheric pressure of CO_2 for 24 h, in a sealed Schlenk tube, unless otherwise noted. [b] in 1, 4-dioxane, at 100 °C. [c] in DMSO. [d] NaO^tBu (1.0 mmol). [e] NaO^tBu (1.4 mmol). [f] NaO^tBu (1.2 mmol), **1a** (0.34 mmol). [g] NaO^tBu (1.2 mmol), **1a** (0.4 mmol). [h] CO_2 (2.5 MPa). Xphos = dicyclohexyl(2',4',6'-triisopropyl-[1,1'-biphenyl]-2-yl)phosphine.

With the optimized reaction conditions in hand, we then explored the scope and limitation of the *N*-tosylhydrazones as shown in Scheme 2. Generally, except the 4-CF_3 substituted analogue (**3g**, 33%), the reaction efficiency was not sensitive to the electronic property of the groups on the phenyl ring of *N*-tosylhydrazones as substrates bearing both electron-donating (**3b-3c**, **3h-3k**, 58-76%) and electron-withdrawing groups (**3d-3f**, 62-71%) worked well to deliver the desired products in moderate to good yields. In the cases of substrates with *ortho*- hindrance on the phenyl, **3h-3j** were isolated in acceptable 58%, 61% and 65% yields, respectively. Notably, some functional groups, such as methyl (**3b, 3h, 3j**), fluoro (**3d**), chloro (**3e**), and cyano (**3f**) groups, which were suitable for potential further functionalization, survived well in these reactions. Importantly, the diversity of the product was further increased as this procedure allow to access 4- (2-naphthyl) (**3l**, 46%), 3- methyl (**3m**, 71%; **3n**, 73%), propyl (**3o**, 70%) and phenyl (**3p**, 56%) of 4-aryl-2-quinolinones in moderate to good yields. In particular, the procedure was applicable to the construction of fused cyclic product. For instance, **3q** could be isolated in 60% yield.



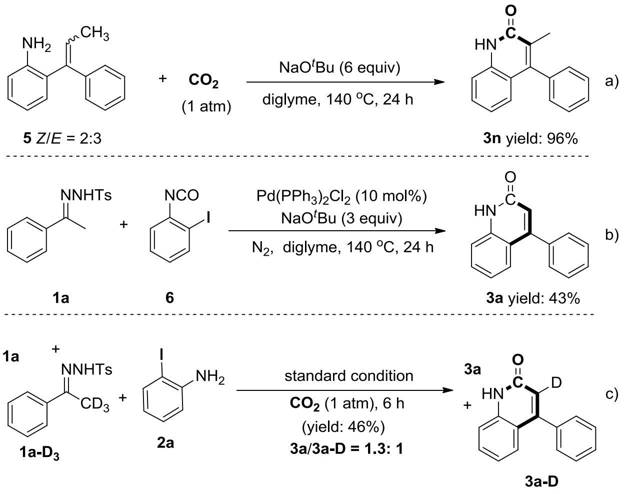
Scheme 2. Scope of *N*-tosylhydrazones. Reaction conditions: **1** (0.34 mmol), **2** (0.2 mmol), $Pd(PPh_3)_2Cl_2$ (0.02 mmol), NaO^tBu (1.2 mmol), diglyme (3.0 mL), a 140 °C under atmospheric pressure of CO_2 for 24 h, in a sealed Schlenk tube.

Next, the scope of 2-iodoanilines was also investigated. A shown in Scheme 3, the substituted 2-iodoanilines proceede smoothly with *N*-tosylhydrazone (**1a**) and atmospheric pressure c CO_2 to afford 4-aryl-2-quinolinones with substituents on the 6-, 7 and 8- position in moderate to good yields. To our delight, th substrates bearing strong electron-withdrawing groups such a cyano, nitro groups also ran smoothly under the standar conditions to afford products **4g** and **4h** in 57% and 45% yields respectively.

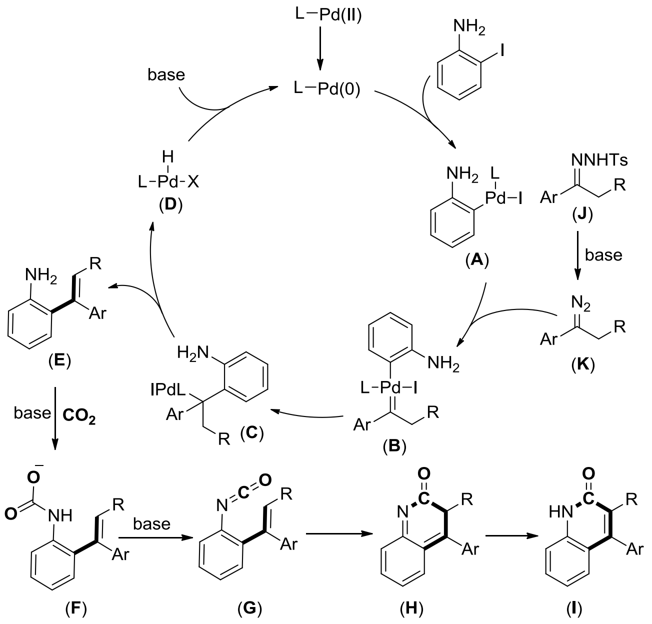


Scheme 3. The substrate scope of 2-iodoanilines. Reaction conditions: **1a** (0.34 mmol), **2** (0.2 mmol), $Pd(PPh_3)_2Cl_2$ (0.02 mmol), NaO^tBu (1.2 mmol), diglyme (3.0 mL), at 140 °C under atmospheric pressure of CO_2 for 24 h, in a sealed Schlenk tube.

Some control experiments were conducted to get some insights into this transformation. Firstly, 2-(1-phenylprop-1-en-1-yl)aniline **5** (*Z*/*E* = 3/2)[14] was employed to react with 1 atm of CO_2 in the presence of 6 equivalents of Na^tOBu and the 3-methyl-4-phenyl-2-quinolinone **3n** was isolated in 96% yield, indicating both *Z* and *E* isomers took part in this transformation (Scheme 4a). This was consistent with the 6π-electrocyclic reaction pathway proceeding with isocyanate intermediate.[15] Subsequently, 1-iodo-2-isocyanatobenzene (**6**) was employed to react with **1a** in the presence of $Pd(PPh_3)_2Cl_2$ (10 mol%) and NaO^tBu (3 equiv) under N_2 in diglyme at 140 °C for 24 h, and **3a** was isolated in 43% yield (Scheme 4b), indicating the possibility of **6** serving as an intermediate (for details, see Supporting Information). Moreover, the intermolecular kinetic isotopic experiment confirmed the k_H/k_D in methyl was 1.3. Thus, the *β*-hydride elimination was not the rate determining step during this transformation (Scheme 4c).



Scheme 4. Mechanism study.



Scheme 5. Proposed mechanism.

Based on these experimental results, a proposed mechanism was outlined in Scheme 5. The reaction is initiated by oxidative addition of Pd^0 to 2-iodoaniline to afford Pd^{II} species **A**. Meanwhile, diazo substrate **K** is generated *in situ* from *N*-tosylhydrazone **J** with the assistance of base. After that, the reaction of **K** and Pd^{II} species **A** affords palladium carbene intermediate **B**. Subsequently, migratory insertion of aromatic group to the carbenic carbon gives intermediate **C**, which encounters *β*-hydride elimination to deliver amine **E** and regenerates the Pd^0 catalyst in the presence of base. Then, with the assistance of base, the amine **E** reacts with CO_2 rapidly, leading to carbamate **F**, which converts to isocyanate intermediate **G** by dehydration.[16] Finally, the 6π- electrocyclic reaction in **G** takes place towards intermediate **H**, which affords 4-aryl-2-quinolinone **I** via [1,5]-σ-hydrogen shift in **H**. Alternatively, before the 6π- electrocyclic reaction of **G**, pathway involving intermediate **6** could not be thoroughly ruled out at the current stage. (for details, see Supporting Information)

In conclusion, we have developed an efficient palladiur catalyzed three-component reaction between *N*-tosylhydrazones 2-iodoanilines and CO_2, allowing to quick access a series of 4 aryl-2-quinolinones in moderate to good yields with diversity. Thi procedure tolerates some functional groups well. This protoco features with simultaneous formation of four novel bonds i.e. tw C–C, one C-C double and one C-N bond in amide, representin an efficient methodology for incorporation of CO_2 int heterocycles.

Experimental Section

Experimental Details: In a glovebox, *N*-tosylhydrazone (0.34 mmol), 2 iodoaniline (0.2 mmol), $Pd(PPh_3)_2Cl_2$ (0.02 mmol, 14 mg), NaO^tBu (1.2 mmo 115.2 mg) and diglyme (3.0 mL) was added into a 20 mL Schlenk tub equipped a Teflon cap. The reaction vessel was evacuated to about -0.1 MP (last 30 seconds per time) and backfilled with CO_2 (1 atm) in three times. Th sealed Schlenk tube was stirred at 140 °C for the desired time. After th reaction mixture was cooled to room temperature, 1 M HCl (1.5 mL) was adde to terminate the reaction. The reaction mixture was diluted with EtOAc an washed with saturated brine water. The organic layer was dried over anhydrou $MgSO_4$ and concentrated in vacuum. The residue was purified by flash colum chromatography on silica gel with CH_2Cl_2-ethyl acetate as eluent to give th desired product.

Acknowledgements

We thank the National Natural Science Foundation of China (nos 21272028 and 21572025), “Innovation & Entrepreneurshi Talents” Introduction Plan of Jiangsu Province, the Key Universit Science Research Project of Jiangsu Province (15KJA150001) Jiangsu Key Laboratory of Advanced Catalytic Material &Technology (BM2012110) and Advanced Catalysis and Gree Manufacturing Collaborative Innovation Center for financia supports. Sun thank the National Natural Science Foundation c China (no. 21602019), Young Natural Science Foundation c Jiangsu Province (BK20150263) for financial supports.

Keywords: carbon dioxide • 4-aryl-2-quinolinone • *N* tosylhydrazone • multi-component reaction

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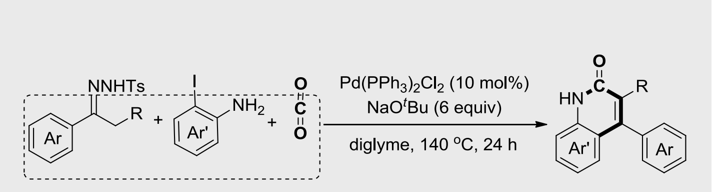

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CO_2 Fixation

*Song Sun, Wei-Ming Hu, Ning Gu, Jiang Cheng** ■■ – ■■

Palladium-Catalyzed Multi-Component Reactions of *N*-Tosylhydrazones, 2-Iodoanilines and CO_2 towards 4-Aryl-2-Quinolinones