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Palladium-Catalyzed C8-Oxygenation of Naphthalene Derivatives: a Direct Access to Naphtholactone Skeleton

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Abstract. Herein, a direct C8-oxygenation of naphthalene derivatives is described. Different carbonyl groups were used as directing group to deliver corresponding naphthols *via* a palladium-catalyzed oxidation reaction using $\text{PhI}(\text{OAc})_2$ in a TFA/TFAA mixture. Interestingly, when Weinreb amide was employed as directing group, the naphtholactone skeleton was directly obtained. This methodology was applied to the synthesis of variously substituted naphtholactones.

Keywords: C–H oxygenation; naphtholactone; 1-carbonylnaphthalene; oxidation; palladium-catalysis

Naphtholactone is an important moiety in chemistry, commonly found in more or less complex natural products (Figure 1).^[1] Many studies have been carried out whether on their biosynthesis or on their biological activities.^[2] For instance, norbadione A is known as a pigment displaying antioxidant properties.^[3] Different synthetic studies have been reported but generally requiring a large number of steps and harsh conditions.^[4]

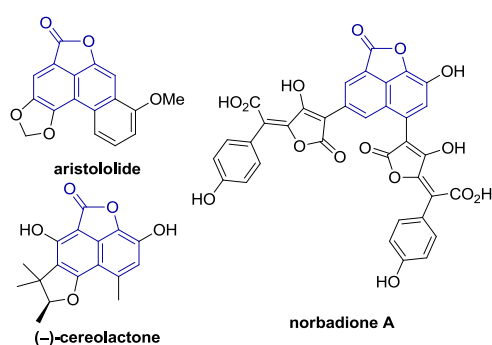
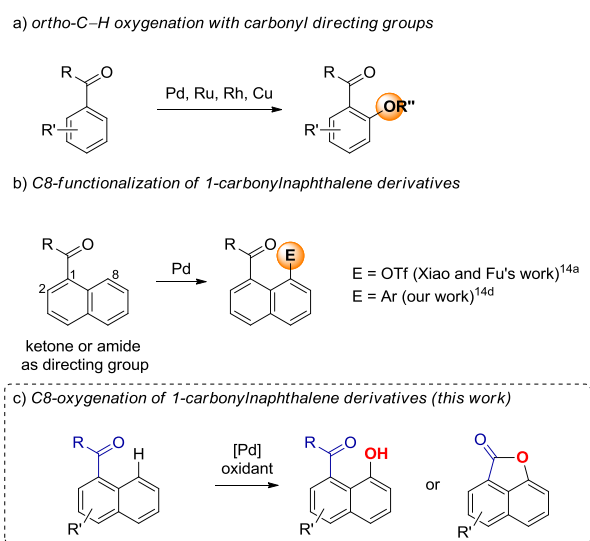


Figure 1. Natural products bearing a naphtholactone moiety

In this context, the development of a direct oxygenation method of naphthalenes to easily obtain naphtholactone is of considerable interest. In order to form the lactone moiety, a suitable strategy would be based on naphthalene oxygenation *via* a carbonyl directed C–H activation of the *peri*-position. Indeed, this strategy would allow the introduction of the oxygen atom of the lactone while using the carbonyl group as directing group and then as lactone precursor.

In recent years, many advances have been made on direct C–H oxygenation of arenes.^[5] The importance of phenols incited chemists to develop straightforward synthetic methods with high atom- and step-economy so that different transition metals combined or not with photochemistry or electrochemistry have been employed to create C–O bonds.^[6] Although, first examples were developed with classical strongly coordinating directing groups,^[7] progress has recently been made with weakly coordinating groups.^[8] Concerning *ortho* C–H hydroxylation of arenes with carbonyl directing groups, different transition metals were reported as efficient catalysts such as Pd,^[9] Ru,^[10] Rh,^[11] or Cu (Scheme 1a).^[12] Regarding the naphthalene core, several C–H functionalization methods have been reported over the last decade.^[13] Nowadays, almost all naphthalene positions can be directly functionalize even if the C2-position remains the most studied. In regards to C8-functionalization with carbonyl as weakly coordinating directing group, few works have been reported.^[14] Inspired by the work of the

group of Xiao and Fu on C8-triflation,^[14a] we recently disclosed a palladium-catalyzed C8-arylation of naphthalene derivatives (Scheme 1b).^[14d] However, C8-functionalization of 1-carbonyl naphthalenes is still under-studied. Another strategy, based on transient directing groups, has been lately developed by Zhang's team.^[15] With this in mind, we were keen to develop a direct C8-oxygenation of naphthalene derivatives using carbonyl moiety as directing group which would allow a fast and easy access to naphtholactone (Scheme 1c).



Scheme 1. (a) Well-established C–H oxygenation of arenes. (b) Previous naphthalene C8-functionalizations with carbonyl directing groups. (c) This work on C8-oxygenation.

Herein we present a palladium-catalyzed C8-oxygenation of 1-amidonaphthalene derivatives which gives the first direct access to naphtholactone skeleton *via* C–H activation.

At the onset of our investigation, we focused our attention on the C8-oxygenation of naphthalen-1-yl(pyrrolidin-1-yl)methanone **1aa**. Pd(OAc)₂ (10 mol%) was employed as catalyst and different oxidants were tested in a mixture of trifluoroacetic acid (TFA)/trifluoroacetic anhydride (TFAA) as solvent (Table 1, entries 1–3). Since pioneering work of Sanford's group,^[16] several research groups reported the use of hypervalent iodine reagents in naphthalene functionalization.^[17] For our reaction, (diacetoxyiodo)benzene was found to be the best oxidant, delivering desired naphthol **2aa** in 57% NMR yield. A full optimisation of the reaction

was carried out,^[18] and 40 °C was found to be the ideal temperature for the reaction with a 75% NMR yield (Table 1, entries 4–6). Finally, the amount of oxidant as well as other palladium sources were evaluated without any improvement (Table 1, entries 7–10). It is important to note that a decrease in the amount of catalyst to 5 mol% showed a small drop in yield (Table 1, entry 11) while, without palladium catalyst no product was formed and only a slight degradation of the starting material was observed (Table 1, entry 12).

Table 1. Selected optimization results^[a]

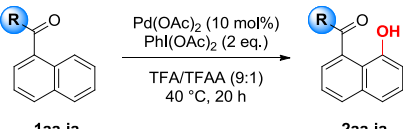

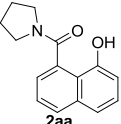

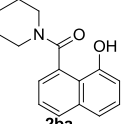

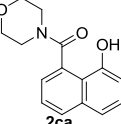

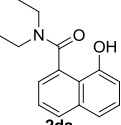

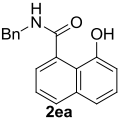

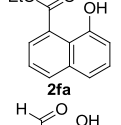

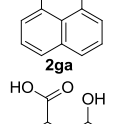

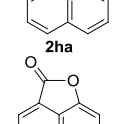

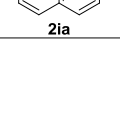
entry	[Pd]	Oxidant (eq.)	T (°C)	Yield (%) ^[b]
1	Pd(OAc) ₂	PhI(OAc) ₂ (2)	80	57
2	Pd(OAc) ₂	Selectfluor (2)	80	33
3	Pd(OAc) ₂	Benzoyl peroxide (2)	80	47
4	Pd(OAc) ₂	PhI(OAc) ₂ (2)	20	47
5	Pd(OAc) ₂	PhI(OAc) ₂ (2)	40	75
6	Pd(OAc) ₂	PhI(OAc) ₂ (2)	60	57
7	Pd(OAc) ₂	PhI(OAc) ₂ (1.5)	40	52
8	Pd(OAc) ₂	PhI(OAc) ₂ (2.5)	40	64
9	Pd(OAc) ₂	PhI(OAc) ₂ (3)	40	58
10	PdCl ₂ (Ph ₃) ₂	PhI(OAc) ₂ (2)	20	30
11	Pd(OAc) ₂ ^[c]	PhI(OAc) ₂ (2)	40	60
12	-	PhI(OAc) ₂ (2)	20	0

[a] Reactions were carried out using **1a** (0.22 mmol), oxidant and [Pd] (0.022 mmol) in TFA/TFAA (0.5 mL, 9:1) for 20 h. [b] NMR yield determined using 1,3,5-trimethoxybenzene as internal standard. [c] 5 mol% of Pd(OAc)₂ was used.

With the optimized conditions in hand, we investigated the scope of the reaction by studying first the influence of the nature of the directing group (Table 2). Cyclic amides were found to be efficient directing groups for the C8-oxygenation. First, (8-hydroxynaphthalen-1-yl)(pyrrolidin-1-yl)methanone **2aa** was isolated in 73% yield, in line with the NMR yield previously observed (Table 2, entry 1). Similarly, piperidine and morpholine amides allowed the formation of corresponding naphthols in good yields (Table 2, entries 2–3). Despite a lower yields, C8-oxygenation was also achieved with a non cyclic amide as well as secondary amide (Table 2,

entries 4-5). Other functional groups, such as ester, aldehyde and carboxylic acid, were tried in the reaction without a lot of success (Table 2, entry 6-8). Ethyl 8-hydroxy-1-naphthoate **2fa** and 8-hydroxy-1-naphthaldehyde **2ga** were isolated in low yields (24% and 29% respectively) whereas no naphthol was observed with a carboxylic acid as directing group. Finally, the reaction was carried with a Weinreb amide as directing group, and very interestingly lactone **2ia** was directly isolated as the only product in very good yield (85%) (Table 2, entry 9).

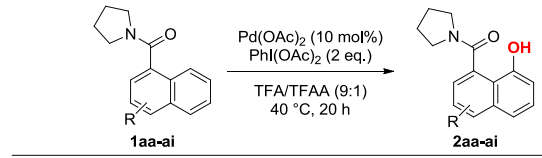
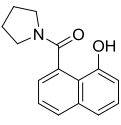
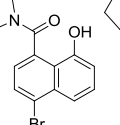
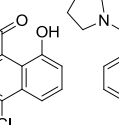
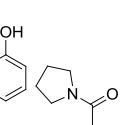
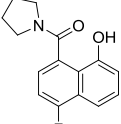
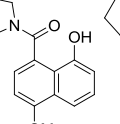
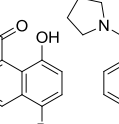
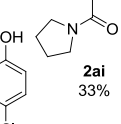
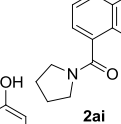
Table 2. Scope of the carbonyl directing group^[a,b]

			
Entry	R	Product	Yield (%)
1			73
2			47
3			52
4			35
5			26
6			24
7			29
8			0
9			85

[a] Reactions were carried out using **1** (0.5 mmol), PhI(OAc)₂ (1 mmol) and Pd(OAc)₂ (0.05 mmol) in trifluoroacetic acid/trifluoroacetic anhydride (1.14 mL, 9:1) for 20 h at 40 °C. [b] Isolated yields were reported.

According to the results on the directing group scope, several naphthalene derivatives with an amidopyrrolidine group were engaged in the oxygenation reaction (Table 3). 1-Amidonaphthalenes **1ab-1ae**, substituted by a halogen atom in position 4, were compatible with the reaction conditions and delivered the corresponding naphthol in moderate to good yields (33-59%). Unfortunately, a methoxy substituent was not tolerated in the reaction and only degradation was observed, probably due to overoxidation products which were unstable in an acidic medium (yield of **2af** = 0%). To extend the scope, naphthalene derivatives with halogen atoms in position 5 **1ag-1ah** were successfully hydroxylated. Finally, 1,4-diamido naphthalene **1ai** was tried under our reaction conditions and mono-oxygenated product **2ai** was isolated as the only product.

Table 3. Scope of the pyrrolidine amide derivatives^[a,b]

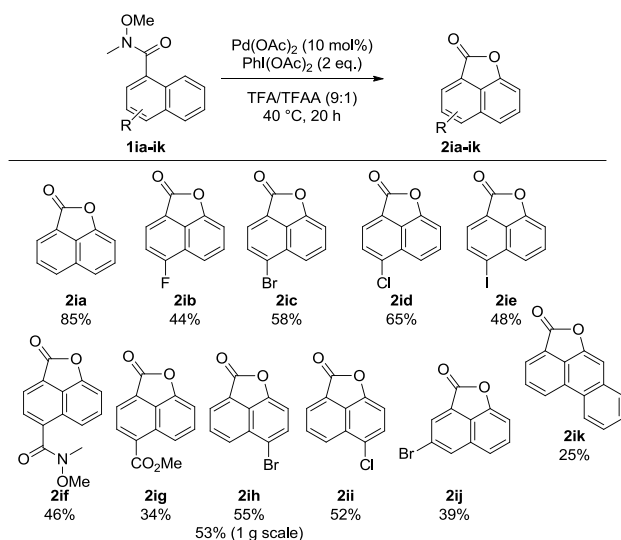
			
			
73%	42%	52%	33%
			
59%	0%	46%	37%
			
33%			

[a] Reactions were carried out using **1a** (0.5 mmol), PhI(OAc)₂ (1 mmol) and Pd(OAc)₂ (0.05 mmol) in trifluoroacetic acid/trifluoroacetic anhydride (1.14 mL, 9:1) for 20 h at 40 °C. [b] Isolated yields were reported.

Following our goal of naphtholactone synthesis for synthetic applications, different Weinreb amide derivatives were tested in the reaction (Table 4). Pleasingly, as previously, naphthalenes bearing a halogen atom in position

4 delivered corresponding lactones **2ib-2ie** in good yields (44-65%). Then, the effect of carbonyl substituents was evaluated. When the reaction was realized with diamide **1if**, monolactone **2if** was obtained in 46% yield. In addition, when naphthalene derivative **1ig** bearing a Weinreb amide and an ester group was engaged in the reaction, only the Weinreb amide was an efficient directing group and lactone **2ig** was synthesized. Other substituent positions were possible for the naphthalene derivative. Indeed, to our delight, bromine or chlorine atoms in position 3 or 5 were compatible with the reaction conditions. The reaction was also carried out in gram scale and lactone **2ih** was obtained in a similar yield of 53%. Finally tetracyclic core of aristololide **2ik** was obtained in a very short number of steps. Indeed, amide **1ik** was obtained in one step based on the work of Li and Shi's group on phenanthrene derivative synthesis,^[19] and so our methodology allowed the synthesis of natural product skeleton in only two steps.

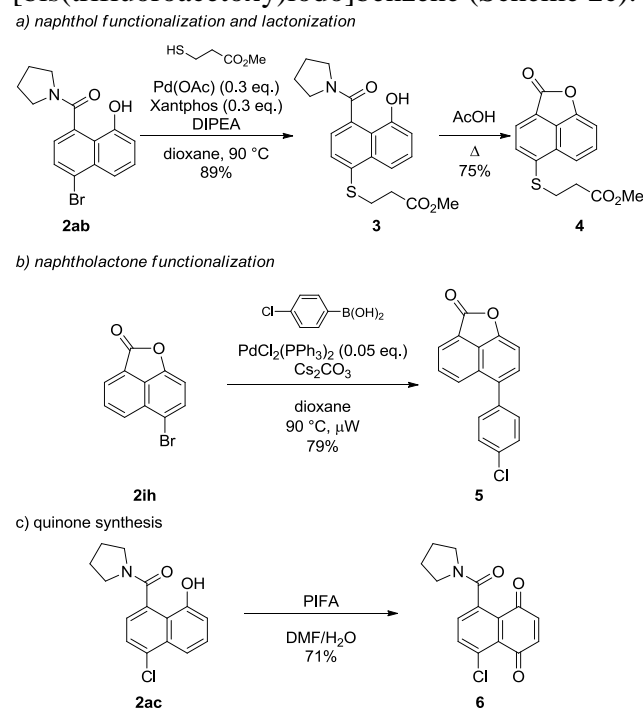
Table 4. Scope of the naphtholactone synthesis^[a,b]



[a] Reactions were carried out using **1i** (0.5 mmol), PhI(OAc)₂ (1 mmol) and Pd(OAc)₂ (0.05 mmol) in trifluoroacetic acid/trifluoroacetic anhydride (1.14 mL, 9:1) for 20 h at 40 °C. [b] Isolated yields were reported.

In order to demonstrate the synthetic utility of our methodology, different transformations were realized with our oxygenated products (Scheme 2). First, although electron donating groups were not compatible with our conditions, bromo-naphthol **2ab** was coupled with methyl 3-

mercaptopropionate to obtain thioether **3** in 89% yield which, after acidic treatment, delivered naphtholactone **4** (Scheme 2a). Next, lactone **2ih** was engaged in a Suzuki-Miyaura coupling reaction to generate naphtholactone **5** in 79% yield bearing an aromatic group in position 5 (Scheme 2b). Therefore, these transformations show that, thanks to palladium coupling reactions, it would be possible to obtain easily complex naphtholactone derivatives. Finally, naphthol **2ac** was oxidized into quinone **6** in 71% yield thanks to [bis(trifluoroacetoxy)iodo]benzene (Scheme 2c).



Scheme 2. Product transformations

In conclusion, we developed a palladium-catalyzed C8-oxygenation of naphthalenes based on a C–H activation approach. Amides were found to be suitable directing groups for this transformation and, in particular, Weinreb amide which directly delivered the corresponding lactone giving very interesting perspectives to apply our strategy for the synthesis of biologically active compounds. Further explorations of C–H activation reactions with naphthalene derivatives are currently in progress in our laboratory.

Experimental Section

General procedure for the C–H oxygenation: Naphthalene **1** (0.5 mmol), Pd(OAc)₂ (11 mg, 0.05 mmol),

0.1 eq.) and $\text{PhI}(\text{OAc})_2$ (322 mg, 1 mmol, 2 eq.) were placed in a tube. TFA (1.01 mL) and TFAA (113 μL) were added and the tube was closed with a screw cap. The mixture was stirred at 40 °C during 20 h. At r.t., the mixture was diluted with CH_2Cl_2 and a saturated aqueous solution of NaHCO_3 was slowly added. The phases were separated and the aqueous phase was extracted with CH_2Cl_2 . The combined organic phases were dried over MgSO_4 and concentrated in vacuo. The residue was purified by flash chromatography.

Acknowledgements

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