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Pd(0)-Catalyzed intramolecular arylative dearomatization of β-naphthols†

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An efficient Pd(0)-catalyzed intramolecular arylative dearomatization of β-naphthols is described. Using Q-Phos as a ligand, the arylative dearomatization reaction proceeded smoothly affording excellent yields and chemoselectivity even when the catalyst loading was reduced to 0.1 mol%. This method offers an efficient access to a series of structurally diverse spirocarbocycles. Preliminary investigation indicates that an enantioselective reaction is feasible in the presence of a chiral phosphoramidite ligand.

Spirocarbocycles often exist in diverse natural products, biologically active molecules[1] and useful chemical structures[2] (Fig. 1). Therefore, it has attracted the intense attention of organic chemists to synthesize the unique structure of spirocarbocycles. Due to the congested quaternary carbon centers existed in spirocarbocycles, it has been a long-standing challenge to develop convenient synthetic methodologies. The recent progress in organometallic catalysis provides new solutions for the synthesis.[3] However, highly efficient synthetic routes to obtain structurally diverse spirocarbocycles from readily available starting materials are still in great demand.

Phenol and its derivatives are important chemical starting materials and are widely used in organic synthesis.[4] The recent development on the dearomatization of phenol and derivatives provides a novel route for the construction of spirocarbocycles.[5,6] Of particular note, Pd-catalyzed cross-coupling type dearomatization reactions of anilines,[7] phenols,[8] indoles,[9] pyrroles,[10] pyridines[11] and furans[12] have been reported to construct interesting and useful structures. We envisaged that through an intramolecular design, spirocarbocycles can be easily obtained *via* the Pd-catalyzed arylative dearomatization of phenol derivatives. However, the chemoselectivity between the *O*-arylation and *C*-arylation reaction pathways is of great challenge in Pd-catalyzed dearomatization reactions especially when a quaternary carbon center is needed to be formed at the *ortho*-position of the phenolic hydroxyl group. For instance, the group of Buchwald reported that only the *O*-arylation product is obtained when an *ortho*-substituted phenol is subjected to Pd(0) catalysis (Scheme 1, eqn (1)).[8b] Recently, Luan and co-workers[8e] reported an elegant microwave-assisted Pd(0)-catalyzed alkyne migratory insertion and β-naphthol dearomatization cascade process, in which excellent chemoselectivity was achieved (Scheme 1, eqn (2)). However, to our knowledge, Pd(0)-catalyzed intramolecular direct arylative dearomatization of β-naphthols has not been reported. Herein, we describe an efficient Pd(0)-catalyzed intramolecular arylative dearomatization of β-naphthols affording all-carbon

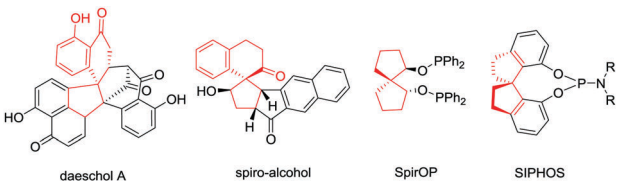
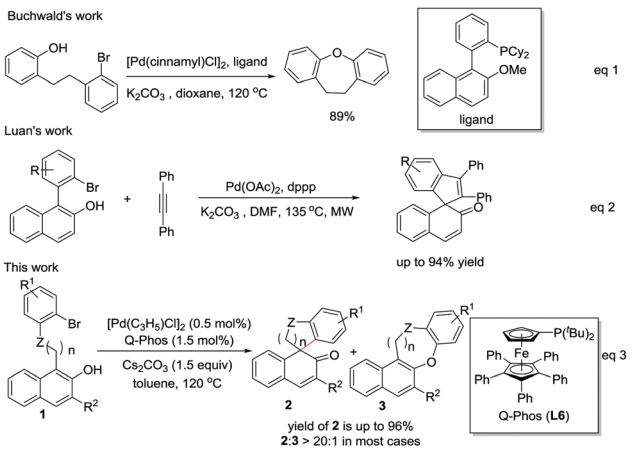


Fig. 1 Biologically active spirocarbocycle-based compounds and ligands.



Scheme 1 Pd-catalyzed arylation of phenols and naphthols.

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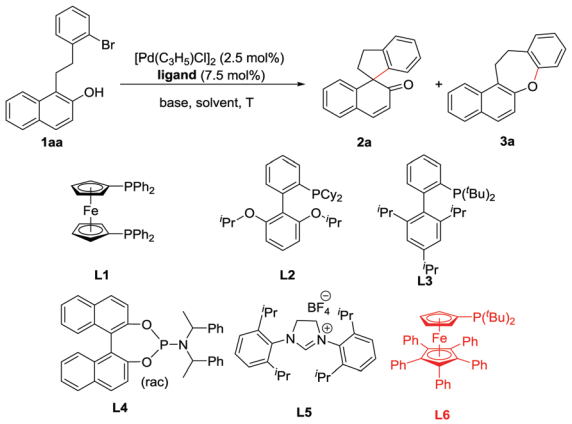
Table 1 Optimization of the reaction conditions[a]



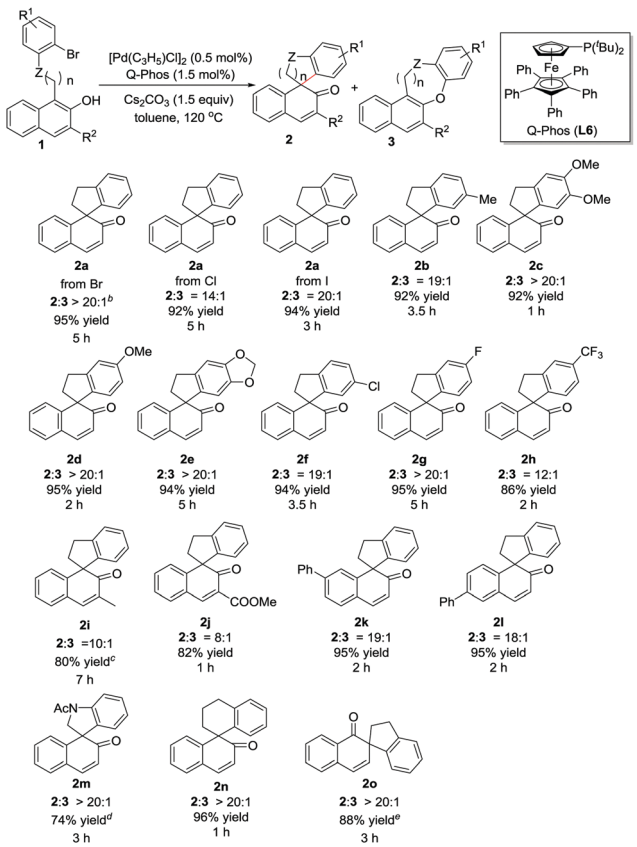
Entry	Ligand	Base	Solvent	T (°C)	Time (h)	**2a** yield[b] (%)	**2a** : **3a**[c]
1	**L1**	K_2CO_3	Toluene	120	8	0	ND
2	PPh_3	K_2CO_3	Toluene	120	8	24	>20 : 1
3	**L2**	K_2CO_3	Toluene	120	8	11[d]	55 : 45
4	**L3**	K_2CO_3	Toluene	120	8	0	<20 : 1
5	**L4**	K_2CO_3	Toluene	120	8	56[d]	>20 : 1
6	**L5**	K_2CO_3	Toluene	120	8	56[d]	>20 : 1
7	**L6**	K_2CO_3	Toluene	120	8	68[d]	4 : 1
8	**L6**	Na_2CO_3	Toluene	120	8	Trace	ND
9	**L6**	K_3PO_4	Toluene	120	8	64	19 : 1
10	**L6**	DBU	Toluene	120	10	13	ND
11	**L6**	Cs_2CO_3	Toluene	120	1	91[d]	19 : 1
12[e]	**L6**	Cs_2CO_3	Toluene	120	5	95[d]	>20 : 1
13[f]	**L6**	Cs_2CO_3	Toluene	120	31	95[d]	>20 : 1
14	**L6**	Cs_2CO_3	Toluene	40	7	92[d]	>20 : 1
15	**L6**	Cs_2CO_3	DCM	40	7	93[d]	>20 : 1
16	**L6**	Cs_2CO_3	THF	40	7	92	>20 : 1
17	**L6**	Cs_2CO_3	CH_3CN	40	7	20	ND
18	**L6**	Cs_2CO_3	Dioxane	40	7	95	>20 : 1

[a] Reaction conditions: **1aa** (0.2 mmol), $[Pd(C_3H_5)Cl]_2$ (0.005 mmol), ligand (0.015 mmol), base (0.3 mmol) in solvent (1.0 mL), T °C. [b] Determined by ^{1}H NMR using CH_2Br_2 (0.2 mmol) as an internal standard. [c] Determined by ^{1}H NMR of the crude products. [d] Isolated yield. [e] $[Pd(C_3H_5)Cl]_2$ (0.5 mol%) and **L6** (1.5 mol%) were used. [f] $[Pd(C_3H_5)Cl]_2$ (0.1 mol%) and **L6** (0.3 mol%) were used.

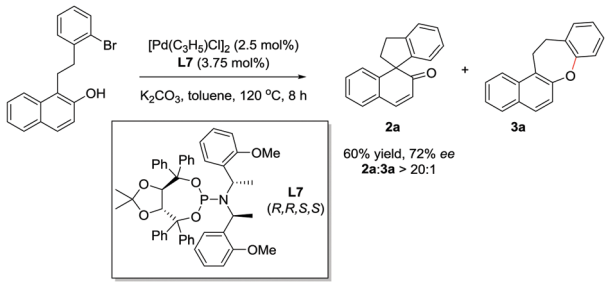
spirocarbocycle structures in excellent yields and chemoselectivity (Scheme 1, eqn (3)).

Initially, 1-(2-bromophenethyl)-2-naphthol (**1aa**) was chosen as a model substrate to examine different ligands under palladium catalysis. The results are summarized in Table 1. No desired dearomatized product was obtained when the diphosphine ligand dppf (**L1**) was used (Table 1, entry 1), while the utilization of PPh_3 led to the formation of the desired dearomatized product (**2a**) in 24% NMR yield (Table 1, entry 2). With the Buchwald-type ligand RuPhos (**L2**), **2a** was isolated in 11% yield together with the isolation of the *O*-arylation product in 9% yield (Table 1, entry 3). When the sterically bulky (di-^{t}Bu)XPhos (**L3**) was used as a ligand, only the *O*-arylation product was obtained (Table 1, entry 4). The utilization of the *rac*-Feringa ligand (**L4**) or SIPr·HBF_4 (**L5**) led to the exclusive formation of **2a** (56% yield, Table 1, entries 5 and 6). With Q-Phos (**L6**) as a ligand, **2a** could be obtained in 68% yield with **3a** isolated in 17% yield. Subsequently, several bases were examined (Table 1, entries 8–11). To our delight, the stronger inorganic base Cs_2CO_3 almost exclusively gave the desired dearomatized product (**2a**) in 91% yield within 1 hour. Lowering the catalyst loading to 0.5 mol% and 0.1 mol% led to slightly higher yields in both cases but a longer reaction time was needed (Table 1, entries 12 and 13). Notably, the reaction underwent smoothly even at 40 °C (Table 1, entry 14). The reaction in varied solvents, such as DCM, dioxane and THF, all gave satisfactory yields at 40 °C (92–95% yields, Table 1, entries 15–18). Considering the efficiency and convenience of the experiments, the optimized conditions were obtained as the following: 0.5 mol% $[Pd(C_3H_5)Cl]_2$, 1.5 mol% Q-Phos (**L6**), and 1.5 equiv. of Cs_2CO_3 in toluene at 120 °C (Table 1, entry 12).

Next, we turned our attention to examine the substrate scope of this reaction. As shown in Scheme 2, substrates by cleaving different C–X bonds all gave their corresponding products in excellent yields and chemoselectivity (X = Cl, 92% yield, **2a** : **3a** = 14 : 1; X = Br, 95% yield, **2a** : **3a** > 20 : 1; X = I, 94% yield, **2a** : **3a** = 20 : 1). Then, a wide range of substituted aryl bromides bearing either electron-donating or electron-withdrawing groups was tested. In all cases, the intramolecular dearomatization reaction proceeded smoothly to afford their corresponding products (**2b–2g**) in excellent yields (92–95%) and chemoselectivity. The structure of **2c** was confirmed by X-ray crystallographic analysis.



Scheme 2 The reaction substrate scope. [a] Reaction conditions: **1** (0.8 mmol), $[Pd(C_3H_5)Cl]_2$ (0.004 mmol), **L6** (0.012 mmol), Cs_2CO_3 (1.2 mmol) in toluene (4.0 mL), 120 °C. Isolated yield. [b] Determined by ^{1}H NMR of the crude products. [c] **1** (0.4 mmol), Cs_2CO_3 (0.6 mmol) in toluene (2.0 mL). [d] **1** (0.2 mmol), Cs_2CO_3 (0.3 mmol) in toluene (1.0 mL), 60 °C. [e] **1** (0.2 mmol), $[Pd(C_3H_5)Cl]_2$ (0.005 mmol), **L6** (0.015 mmol), Cs_2CO_3 (0.3 mmol) in toluene (1.0 mL).

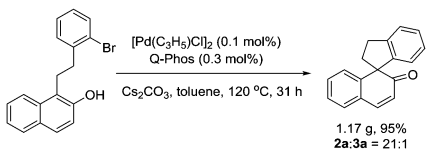


Scheme 3 Pd(0)-Catalyzed asymmetric intramolecular arylative dearomatization of β-naphthols.

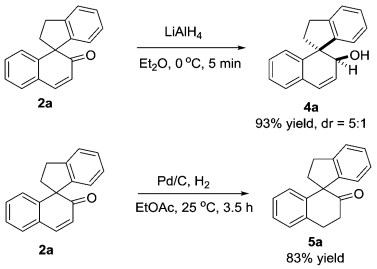
The reaction of the CF_3-substituted substrate gave the desired product (**2h**) in good yield with slightly decreased chemoselectivity (86% yield, **2h**:**3h** = 12:1). Then the influence of the substituent group on the β-naphthol moiety was investigated. 3-Substituted (Me, CO_2Me) naphthols were transformed into dearomatized products (**2i** and **2j**) in good yields and chemoselectivity (80% yield, **2i**:**3i** = 10:1; 82% yield, **2j**:**3j** = 8:1). Fortunately, the substituents on the other positions (6-Ph and 7-Ph) of the β-naphthol moiety did not influence the chemoselectivity to give **2k** and **2l** in excellent yields and chemoselectivity (95% yield, **2k**:**3k** = 19:1; 95% yield, **2l**:**3l** = 18:1). Apart from substrates bearing an all-carbon tether, the substrate with an *N*-linked tether (**1m**) was also compatible, affording the desired product (**2m**) in 74% yield and excellent chemoselectivity (**2m**:**3m** > 20:1) under relatively milder conditions (at 60 °C). For substrate **1n** with an extended carbon chain, the desired dearomatized product with a 6-membered ring formation (**2n**) was obtained smoothly in 96% yield and excellent chemoselectivity (**2n**:**3n** > 20:1). Interestingly, the reaction of the α-naphthol substrate gave the dearomatized product (**2o**) in 88% yield with a **2o**:**3o** ratio of 20:1.

The asymmetric reaction was also explored. Several commercially available chiral phosphine ligands were screened. TADDOL-derived phosphoramidite (**L7**) was found to be the optimal ligand. In the presence of 2.5 mol% $[Pd(C_3H_5)Cl]_2$ and 3.75 mol% **L7**, the reaction of **1aa** could give **2a** in 60% yield and 72% ee with excellent chemoselectivity (**2a**:**3a** > 20:1, Scheme 3, see the ESI† for details).

To further demonstrate the utility of this method, a gram-scale reaction and several transformations of the 2-naphthalenone product have been carried out. The intramolecular dearomatization of **1aa** on a 5.0 mmol scale gave the desired product **2a** in 95% yield and excellent chemoselectivity (**2a**:**3a** = 21:1) while the catalyst loading could be further reduced to 0.1 mol% (Scheme 4). In addition, the ketone group of the product (**2a**) could be



Scheme 4 Gram-scale reaction.



Scheme 5 Transformations of the 2-naphthalenone product.

selectively reduced by $LiAlH_4$ to afford the alcohol **4a** in 93% yield (dr = 5:1), while the carbon–carbon double bond could be reduced by Pd/C catalyzed hydrogenation to afford **5a** in 83% yield (Scheme 5).

In summary, we have developed a Pd-catalyzed intramolecular dearomatization of β-naphthols to construct an all-carbon quaternary stereocenter at the *ortho*-position of the hydroxyl group with excellent yields and chemoselectivity. A series of structurally diverse spirocarbocycles were obtained efficiently and conveniently. Further studies on the application of the current method and development of more efficient catalytic asymmetric reactions are currently underway in our laboratory.

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