Organic LETTERS

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# Diastereospecific and Enantioselective Access to Dispirooxindoles from Furfurylcyclobutanols by Means of a Pd-Catalyzed Arylative Dearomatization/Ring Expansion Cascade

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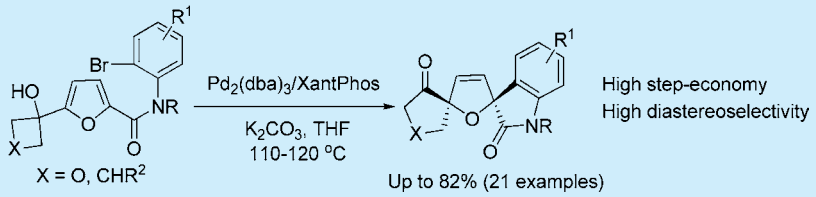
Supporting Information

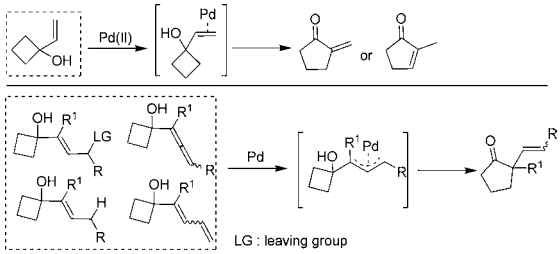



**ABSTRACT:** We report a Pd-catalyzed arylative dearomatization/ring expansion cascade of furfurylcyclobutanols that involves a spiro $\pi$-allyl palladium intermediate and affords structurally novel dispirooxindoles containing two quaternary carbon centers in good yields with high step economy, diastereospecificity, and enantioselectivity.

Many natural products and bioactive compounds have dispirocycle skeletons.[1] The development of step-economic methods for highly stereoselective access to dispirocycles with two quaternary carbon centers is of great interest in current organic synthesis. Spirooxindole units are of particular value because these heterocyclic motifs constitute the structural core of a large family of medicinal agents that demonstrate strong cytotoxicity to the A431 human epidermoid carcinoma cell line,[2] the T24 and RT4 human bladder cancer cell lines,[3] lung adenocarcinoma (A549) cells,[4] hepatocellular carcinoma (HepG2) cells,[5] and other cell types.[6] Although enormous effort has been devoted to the development of efficient methods for the synthesis of structurally diverse spirooxindoles, including cycloaddition,[7] addition−cyclization,[8] oxidative rearrangement,[9] intramolecular Heck reaction,[10] and other methods,[11] methods affording access to structurally novel dispirooxindoles with high step economy and stereoselectivity are rare.

Palladium-catalyzed ring expansion of 1-alkenyl cyclobutanols to cyclopentanones via 1,2-carbon shifts is a well-known method for the synthesis of interesting natural products with five-membered-ring frameworks.[12] These reactions take place by initial electrophilic activation of a terminal alkene coordinated to a palladium(II) species to form an alkene complex and subsequent migration of a carbon.[13] Another general method for promoting ring expansion reactions of 1-alkenyl cyclobutanols involves generation of a $\pi$-allyl palladium intermediate in various ways (Scheme 1).[14] Despite the existence of methods for Pd-catalyzed ring expansion of 1-alkenyl cyclobutanols, only structurally simple palladium intermediates have been reported, and therefore there is great demand for methods that can be extended to complex spiro $\pi$-allyl palladium species to stereoselectively furnish dispirocycle frameworks with cyclopentanone moieties and two quaternary carbon centers.[15]

**Scheme 1. Palladium-Catalyzed Ring Expansion of 1-Alkenyl Cyclobutanols**




Recently, we reported a Pd-catalyzed dearomatizing $\alpha$-arylation of furan-2-carboxylic acid (2-bromophenyl)amide **1** to generate spiro $\pi$-allyl palladium intermediate **2** via a Heck insertion of the furan diene (Scheme 2). Intermediate **2** can be stereoselectively transformed into structurally novel spirooxindole **3** or **4** (and **5**) via $\beta$-H elimination or *O*-allylation of the $\pi$-allyl palladium species, respectively.[16] In light of our ongoing interest in dearomatizing transformations of furans,[17] we speculated that if the R substituent on the furan was a 1-hydroxycyclobutyl group, Pd-catalyzed ring expansion might occur to give access to biologically interesting dispirooxindoles (Scheme 2).

We investigated this possibility by carrying out reactions of model substrate **1a** (Table 1). In the presence of 5 mol % $Pd_2(dba)_3$, 10 mol % bis[2-(diphenylphosphino)phenyl]ether (**L1**), and 200 mol % $K_2CO_3$, reaction of **1a** in THF at 100 °C

**Received:** November 7, 2016
**Published:** December 5, 2016





DOI: 10.1021/acs.orglett.6b03339
*Org. Lett.* 2016, 18, 6440−6443

**Scheme 2. Transformations of Spiro π-Allyl Palladium Intermediate 2**

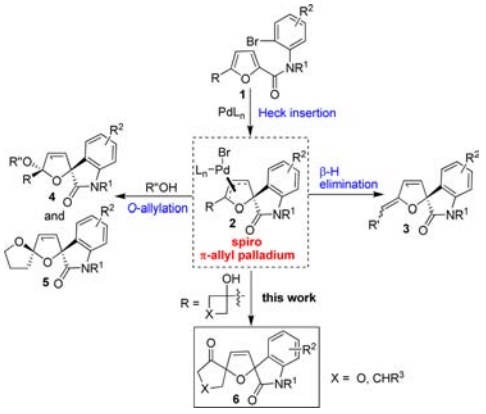
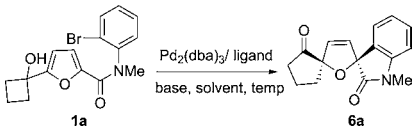



**Table 1. Optimization of Reaction Conditions**[a]



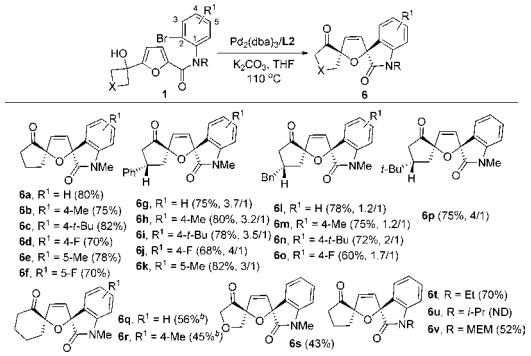

| entry | ligand | base | solvent | yield of **6a** (%)[b] |
|---|---|---|---|---|
| 1 | **L1** | $K_2CO_3$ | THF | 70 |
| 2 | **L2** | $K_2CO_3$ | THF | 75 |
| 3 | **L3** | $K_2CO_3$ | THF | trace |
| 4 | **L4** | $K_2CO_3$ | THF | 35 |
| 5 | $PPh_3$ | $K_2CO_3$ | THF | 67 |
| 6 | DPPP | $K_2CO_3$ | THF | 60 |
| 7 | DPPB | $K_2CO_3$ | THF | 30 |
| 8 | DPPF | $K_2CO_3$ | THF | trace |
| 9 | BINAP | $K_2CO_3$ | THF | 53 |
| 10 | **L2** | LiO*t*Bu | THF | ND |
| 11 | **L2** | $Ag_2CO_3$ | THF | 45 |
| 12 | **L2** | $Na_2CO_3$ | THF | 50 |
| 13 | **L2** | $Li_2CO_3$ | THF | 30 |
| 14 | **L2** | $K_3PO_4$ | THF | 65 |
| 15 | **L2** | KOAc | THF | 35 |
| 16 | **L2** | $K_2CO_3$ | $Et_2O$ | 30 |
| 17 | **L2** | $K_2CO_3$ | MTBE | 65 |
| 18 | **L2** | $K_2CO_3$ | 1,4-dioxane | 72 |
| 19 | **L2** | $K_2CO_3$ | toluene | 70 |
| 20 | **L2** | $K_2CO_3$ | DCE | 60 |
| 21 | **L2** | $K_2CO_3$ | DMF | 65 |
| 22 | **L2** | $K_2CO_3$ | DMSO | 40 |
| 23[c] | **L2** | $K_2CO_3$ | THF | 84 |
| 24[d] | **L2** | $K_2CO_3$ | THF | 73 |

[a]Reaction conditions, unless otherwise noted: **1a** (0.3 mmol), $Pd_2(dba)_3$ (5 mol %), ligand (10 mol %), base (200 mol %), solvent (3 mL), 100 °C, $N_2$ atmosphere, 16 h. **L1** = bis[2-(diphenylphosphino)phenyl]ether, **L2** = 4,5-bis(diphenylphosphino)-9,9-dimethylxanthene, **L3** = $(o\text{-}CH_3Ph)_3P$, **L4** = $(o\text{-}OCH_3Ph)_3P$. NR = no reaction; ND = not detected. [b]Yields were determined by $^1H$ NMR spectroscopy with mesitylene as an internal standard. [c]Temp = 110 °C. [d]Temp = 120 °C.

for 16 h gave desired product **6a** in 70% yield as a single diastereoisomer (entry 1). The stereochemistry of **6a** was assigned based on single-crystal X-ray analysis of **6j** (see the Supporting Information (SI), CCDC 1509629). Examination of several ligands (entries 2−9) revealed that the XantPhos ligand (**L2**) was optimal, giving **6a** in 75% yield. When the strong base LiO*t*Bu was used (entry 10), **6a** was not detected. The use of $Ag_2CO_3$, $Na_2CO_3$, $Li_2CO_3$, $K_3PO_4$, or KOAc as a base (entries 11−15) resulted in lower yields. Screening of various solvents revealed that THF was optimal (entries 16−22). Investigation of reaction temperature (entries 23 and 24) revealed that 110 °C gave the best result, affording **6a** in 84% yield.

To investigate the scope of the reaction, we subjected various *N*-(2-bromophenyl)-2-furancarboxamides **1** to the optimized reaction conditions (Table 1, entry 23) to synthesize various dispirooxindoles **6** (Scheme 3). The reaction had a broad

**Scheme 3. Substrate Scope**[a]




[a]Reaction conditions, unless otherwise noted: **1** (0.3 mmol), $Pd_2(dba)_3$ (5 mol %), **L2** (10 mol %), $K_2CO_3$ (200 mol %), THF (3 mL), temp = 110 °C, under $N_2$, 16 h. Yields are isolated yields dr values were determined by $^1H$ NMR spectroscopy. ND = not detected. [b]Temp = 120 °C.

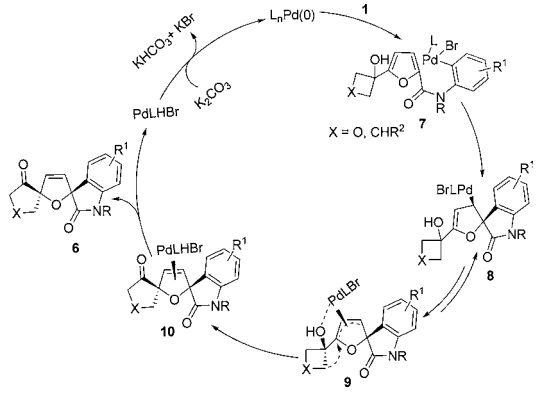
substrate scope; in most cases, the expected spirooxindoles **6** were obtained in moderate to good yields and diastereospecificities at the two quaternary carbon centers. The $R^1$ substituent could be a H atom, an electron-donating group (4-Me, 4-*t*-Bu, 5-Me), or an electron-withdrawing group (4-F, 5-F). Notably, when the cyclobutanol moiety was substituted with a phenyl group (**6g−k**), a 3-benzyl group (**6l−o**), or a 3-*tert*-butyl group (**6p**), the corresponding products were obtained in good yields (60−82%) with moderate diastereoselectivity (1.2:1−4:1). When the cyclobutanol moiety was replaced with a cyclopentanol or 3-oxacyclobutanol moiety, the reactions also proceeded well and gave the corresponding products (**6q−r** and **6s**, respectively) in moderate yields. For **6q** and **6r**, a higher temperature (120 °C) was required due to the less ring strain of the five-membered ring than that of four-membered rings. R could be a small alkyl group (Me and Et), but a substrate with a bulky *i*-Pr group did not yield expected product **6u**, possibly owing to steric hindrance. A substrate in which the nitrogen atom was protected with a removable MEM group was also acceptable, affording **6v** in 52% yield.

To explain the diastereospecificity of the formation of the two quaternary carbon centers of **6**, we propose the mechanism shown in Scheme 4. An oxidative addition reaction between Pd(0) and **1** produces palladium complex **7**. Complex **7** undergoes a Heck-type insertion into the furan diene to produce alkyl palladium intermediate **8**, in which the palladium is located on the same side of the furan ring as the phenyl group. Intermediate **8** then isomerizes to spiro allylic palladium intermediate **9**, in which the hydroxyl group is coordinated with the palladium center on the same face of the molecule. Intermediate **9** subsequently undergoes a 1,2-alkyl shift on the



DOI: 10.1021/acs.orglett.6b03339
*Org. Lett.* 2016, 18, 6440−6443

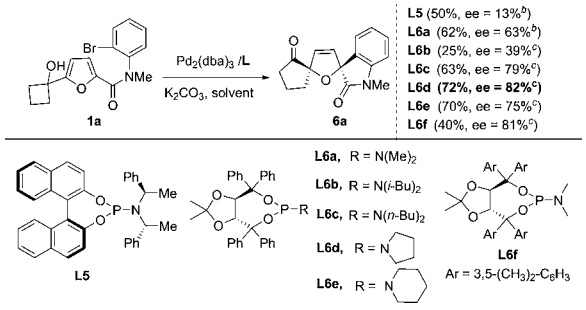
**Scheme 4. Proposed Mechanism for Formation of 6**



face of the molecule opposite the palladium atom (and the phenyl group) to generate intermediate **10**, which is then converted to product **6** with release of the palladium to complete the catalytic cycle.

We also explored an enantioselective version of the protocol with a series of chiral ligands (Scheme 5; for details on optimization of the reaction conditions, see the SI). Use of BINOL-based phosphoramidite **L5** as the ligand led to the formation of **6a** in 50% isolated yield and a low ee (13%). To our delight, when TADDOL-based phosphoramidite **L6a** was used as the chiral ligand, both the yield and ee improved, to 62% and 63%, respectively. Screening of phosphoramidite ligands with various R groups (**L6b**–**L6e**) revealed **L6d** to be the best, providing **6a** in 72% yield with 82% ee. The bulkier ligand **L6f** gave high enantioselectivity (81% ee) but a lower yield (40%). Using **L6d** as the ligand, we carried out reactions of four substrates to synthesize a small library of enantioenriched products (**6a**–**6c**, **6e**–**6f**) in good yields (68–75%) with good ee values (81–83%, Scheme 6).

**Scheme 5. Asymmetric Spirocyclization of 1a**[a]

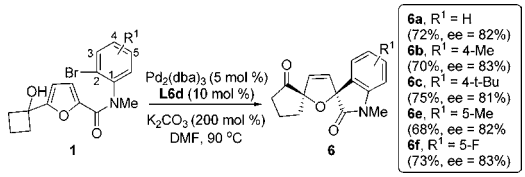


[a]Reaction conditions, unless otherwise noted: **1a** (0.2 mmol), $Pd_2(dba)_3$ (5 mol %), **L** (10 mol %), $K_2CO_3$ (200 mol %), solvent (2 mL), $N_2$ atmosphere, 16 h. Yields are isolated yields ee values were determined by HPLC. TADDOL = α,α,α′,α′-tetraaryl-1,3-dioxolan-4,5-dimethanol. [b]THF was the solvent, temp = 110 °C. [c]DMF was the solvent, temp = 90 °C.

Compound **6** has carbonyl and alkenyl functional groups, which are amenable to numerous further transformations to prepare structurally diverse dispirooxindoles. For example, we carried out hydrogenation of the double bond and reduction of the carbonyl group to afford the corresponding products (**6ga** and **6gb**, respectively) in good yields (Scheme 7).

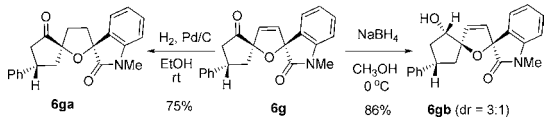
In summary, we report a novel palladium-catalyzed Heck-type arylative dearomatization/ring expansion cascade reaction

**Scheme 6. Asymmetric Synthesis of 6**[a]



[a]Reaction conditions, unless otherwise noted: **1** (0.2 mmol), $Pd_2(dba)_3$ (5 mol %), **L6d** (10 mol %), $K_2CO_3$ (200 mol %), DMF (2 mL), $N_2$ atmosphere, 16 h. Yields are isolated yields, and ee values were determined by HPLC.

**Scheme 7. Hydrogenation and Reduction of 6g**



of furfurylcyclobutanols that provides access to biologically interesting dispirooxindoles with high step economy. Various dispirooxindoles were synthesized in good yields with diastereospecific and enantioselective construction of two quaternary carbon centers. This is the first report of the synthesis of dispiro compounds involving a spiro π-allyl palladium intermediate and a ring expansion strategy, and our results can be expected to pave the way for the design of new reactions aimed at the synthesis of dispiro compounds. Further exploration of the reaction scope and investigation of the bioactivities of the obtained spirooxindoles are underway in our laboratory.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b03339.

Crystallographic data for **6j** (CIF)
Full experimental details including $^1H$, $^{13}C$, and $^1H-^1H$ COSY spectra of **7g**; NOESY spectra of **7g** and **S3g**; HPLC data; X-ray data for **6j** (CCDC 1509629) (PDF)

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### Notes

The authors declare no competing financial interest.

## ■ ACKNOWLEDGMENTS

This work was supported by grants from the National Natural Science Foundation of China (Nos. 21272078 and 21572068), the National Program on Key Research Project (2016YFA0602900), and the Science and Technology Planning Project of Guangdong Province, China (No. 2014A020221035). We thank Chem-Stone Co. for supplying 3-oxetanone.



DOI: 10.1021/acs.orglett.6b03339
Org. Lett. 2016, 18, 6440–6443

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DOI: 10.1021/acs.orglett.6b03339
*Org. Lett.* 2016, 18, 6440−6443