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Hoteite, L., Allen, B.D.W., Elhadj, E.A. et al. (2 more authors) (2024) A Pd-catalyzed annulation strategy to linearly fused functionalized N-heterocycles. Chemistry – A European Journal, 30 (21). e202400116. ISSN 0947-6539

<https://doi.org/10.1002/chem.202400116>

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Hot Paper

A Pd-Catalyzed Annulation Strategy to Linearly Fused Functionalized N-Heterocycles

Larry Hoteite,^[a] Benjamin D. W. Allen,^[a] Ms. Ergaiya A. Elhaji,^[a] Anthony J. H. M. Meijer,^{*,[a]} and Joseph P. A. Harrity^{*,[a]}

Linearly fused polycyclic piperidines represent common substructures in natural products and biologically active small molecules. We have devised a Pd-catalyzed annulation strategy to these compounds that converts readily available 2-tetralones and indanones into these scaffolds with the potential for

control of both enantio- and diastereoselectivity. Importantly, these compounds can be chemoselectively functionalized, providing an efficient and robust methodology to these important nitrogen-containing molecules.

Introduction

Nitrogen-containing molecules are valuable building blocks for pharmaceuticals and agrochemicals. Almost two-thirds of FDA-approved small-molecule drugs contain a nitrogen heterocycle, among which piperidine is the most commonly used.^[1] Over the past decade, molecules that are rich in sp^3 carbon atoms, particularly sp^3 -rich heterocycles, have received increasing interest from the drug discovery community. It has been shown that the fraction of sp^3 hybridized carbon atoms in drug candidates is correlated to important physicochemical properties. Furthermore, it has been demonstrated that sp^3 -rich compounds are more likely to successfully progress through clinical trials and that the presence of chiral stereogenic atoms was found to positively impact the clinical progression of a drug-like molecule.^[2,3]

There are a number of natural products and bioactive compounds that contain a piperidine ring fused to a carbon framework, and these offer a broad range of biological activities. For example, LY266111 is a steroid 5 α -reductase inhibitor, the haouamine alkaloids have anticancer properties, and lysergic acid is a common precursor to a variety of naturally occurring lysergamides which possess potent psychoactive properties (Figure 1).^[4]

We recently reported the synthesis of a variety of architecturally distinct piperidines using a novel Pd-stabilized zwitterion.^[5,6] The use of such dipolar intermediates in synthesis

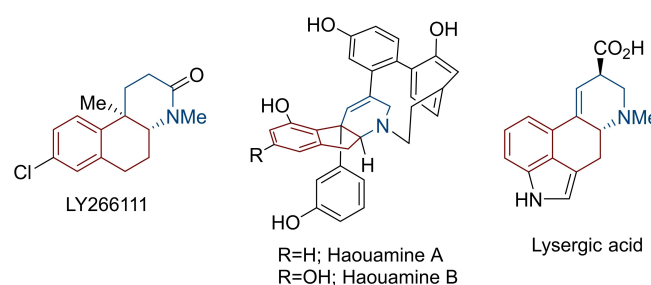
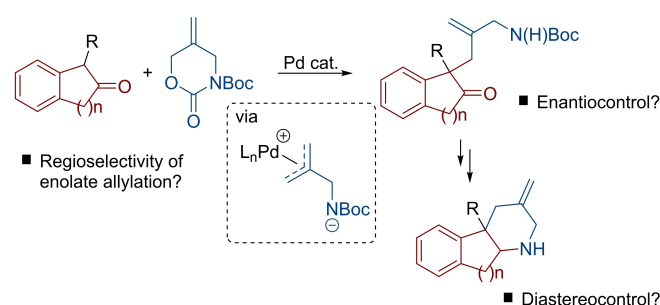


Figure 1. Representative linearly fused N-heterocycles.

is an emerging field^[7] which offers a powerful technology to access highly functionalized products. With regard to fused N-heterocycles, and as shown in Scheme 1, we envisaged that this general strategy could provide an effective route to this class of heterocyclic products, delivering compounds with orthogonal functionality that could undergo further derivatization with the potential to form sp^3 -rich products in a stereocontrolled manner. Herein we report the synthesis of densely functionalized fused piperidines via a Pd-catalyzed allylation/condensation sequence.



Scheme 1. Proposed Pd-catalyzed synthesis of fused piperidines.

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Supporting information for this article is available on the WWW under <https://doi.org/10.1002/chem.202400116>

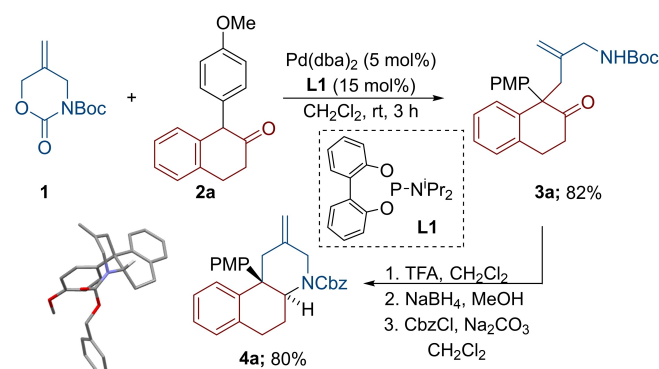
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Results and Discussion

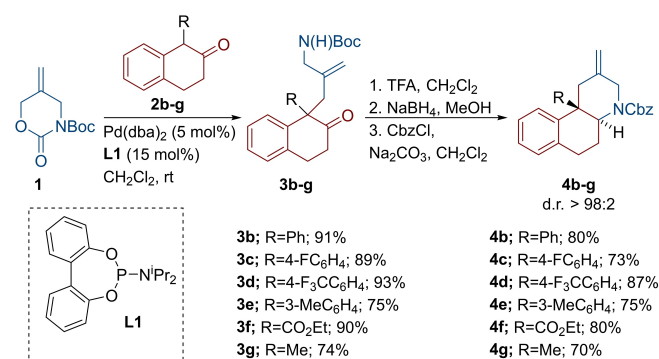
We began by exploring the transformation of 1-(4-methoxyphenyl)-2-tetralone **2a** to the corresponding piperidine derivative (see Scheme 2). We were pleased to find that the Pd-catalyzed allylation step proceeded smoothly and with complete regioselectivity to provide **3a** in high yield. Treatment of **3a** with TFA followed by reduction of the cyclic imine with NaBH₄ led to the desired saturated heterocycle, however, isolation of the free piperidine proved difficult, leading to low product yields. We found that this could be overcome through an in situ protection of the amine with a Cbz group prior to purification. Overall, this sequence allowed **4a** to be isolated in good overall yield as a single diastereomer which was assigned as trans by X-ray crystallography.

Extending the scope of this procedure to a variety of other 1-substituted 2-tetralones proved successful and a range of linearly fused tricyclic piperidines were accessed in good overall yields (Scheme 3). In all cases, the allylation was regioselective for generation of the quaternary substituted products, even in the case of α -methyl tetralone derivative **2g**. The chemistry proved to be tolerant of a range of aryl groups, and in all cases, the products were isolated as a single diastereomer which was assigned as trans by analogy to compound **4a**.^[8]

We next turned our attention to the annulation of the corresponding 2-indanones. These substrates have the potential to form indene enolates either side of the carbonyl, and so we were particularly interested in allylation regiochemistry in this



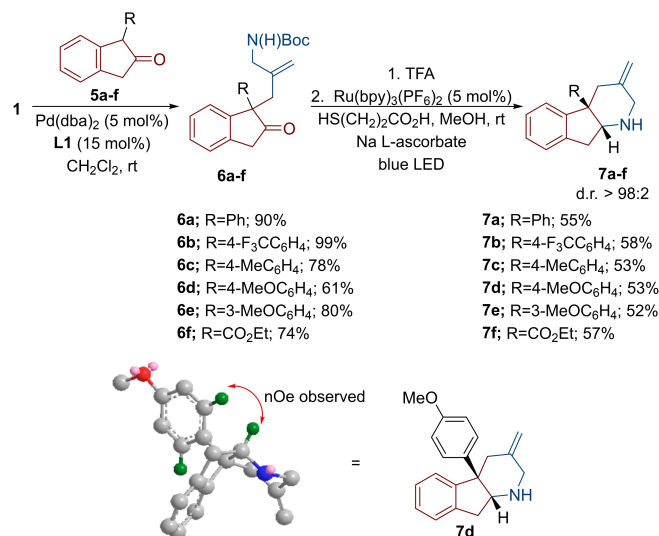
Scheme 2. Initial investigations into the annulation of 1-aryl-2-tetralones.



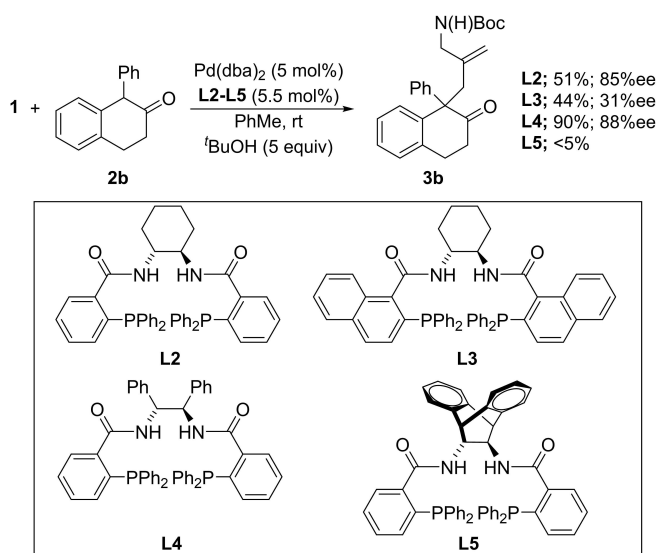
Scheme 3. Synthesis of linearly fused tricyclic piperidines.

case. In the event, a range of indanone-based substrates were subjected to the Pd-catalyzed allylation reaction and provided the corresponding products in high yields and as single regioisomers favoring the quaternary carbon substituted products. In a similar fashion as described for the tetralone-based allylation substrates, we envisioned that a TFA-mediated deprotection-condensation sequence followed by a reduction step would allow rapid access to densely functionalized *N*-heterocycles. However, despite screening a range of hydride reducing agents, we were unable to access the corresponding piperidines, and complex mixtures were instead returned. While the reasons for the inefficiency of this reduction are unclear, this observation prompted us to consider a single electron reduction strategy instead. Therefore, compounds **6a-f** were first exposed to TFA then subjected to the visible-light photo-redox promoted reduction reported by Wenger and co-workers.^[9] Pleasingly, this approach enabled the formation of highly substituted piperidines with contiguous quaternary and tertiary stereogenic centers as single diastereomers and in acceptable yields over two steps. The product stereochemistry was assigned as *cis* by analogy to compound **7d**, and on the basis of a positive nOe correlation between the CH proton (adjacent to the *N*-atom) and the aromatic protons (Scheme 4).

Having established the scope of the general transformation, we next explored the potential for these products to be accessed with high enantiocontrol. We have recently found that the Trost ligand series is effective in promoting enantioselective additions to the Pd π -allyl intermediate derived from **1**^[5c,d] and opted to investigate the enantioselective allylation with this catalyst system. Employing optimal conditions previously developed by us on a related system,^[5c] we screened Trost ligands **L2-L5** and our results are shown in Scheme 5. **L2** generated **3b** with a promising level of enantiocontrol, albeit in modest yield. Naphthyl analog **L3** proved to be detrimental with respect to yield and enantioselectivity, whereas **L4** provided **3b** with high yield and ee. Finally, **L5** bearing a bulky diamine moiety failed to promote the transformation.^[10]



Scheme 4. Synthesis of fused 6-5-6 tricyclic piperidines.

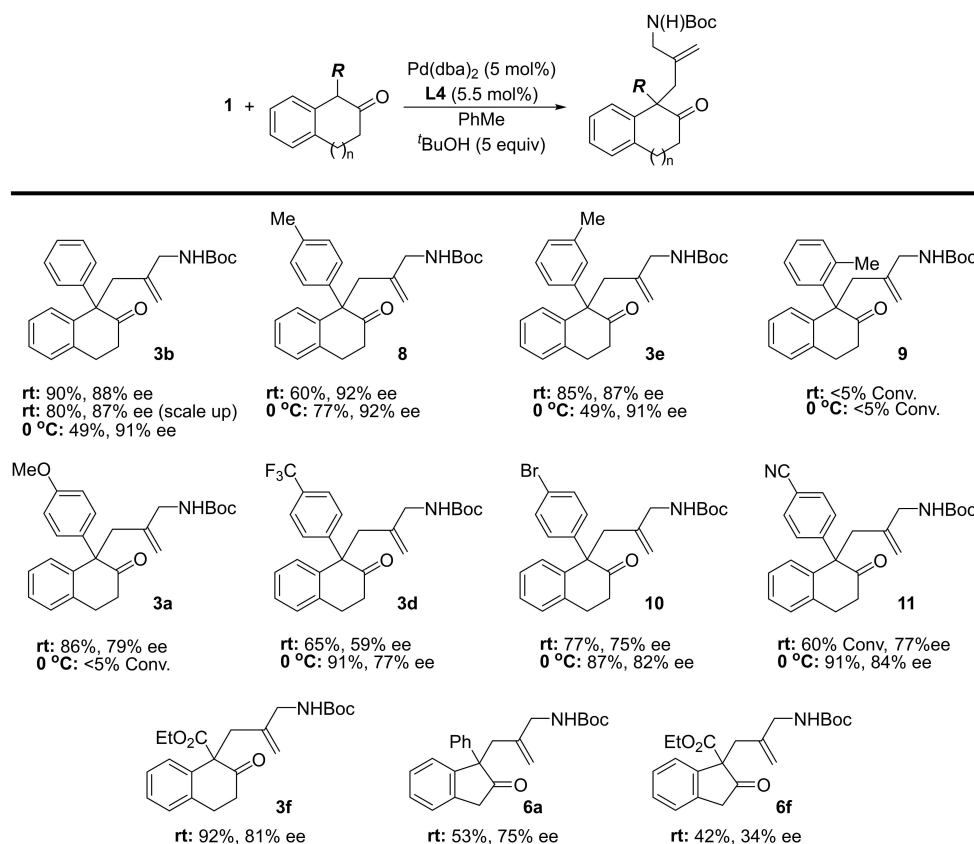
Scheme 5. Ligand screen in the enantioselective allylic alkylation of **2b**.

Having identified an optimal chiral catalyst we next explored the scope of the reaction and our results are summarized in Scheme 6. The synthesis of **3b** could be successfully conducted on 0.5 mmol scale without significant reduction of yield or enantioselectivity. Similar levels of enantiocontrol were observed for *p*- and *m*-Me analogs **8** and **3e**, although the *o*-

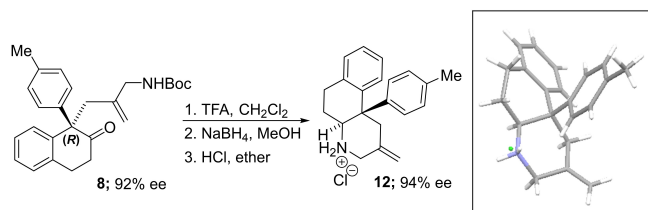
tolyl substituted tetralone was significantly less reactive and the allylic alkylation product **9** was not observed in this case. The use of *p*-electron withdrawing groups generally led to lower levels of enantiocontrol, however, the higher reactivity of these substrates meant that useful yields and ee values could be obtained at 0 °C. In contrast, the substrate bearing a *p*-MeO substituent reacted at room temperature to provide **3a** with modest levels of enantiocontrol. Finally, we found that the corresponding β -keto ester also participated in the asymmetric allylic alkylation as did the corresponding 2-indanones, although the latter gave disappointing yields and enantioselectivities.

We next attempted to determine the stereochemistry of the major enantiomer from the asymmetric allylation step but unfortunately, we were unable to generate suitable crystals for any of the products in Scheme 6. Accordingly, we opted to progress **8** to the corresponding piperidine as the HCl salt **12** (Scheme 7). Pleasingly, this did provide a suitable sample for crystallographic analysis and the configuration was determined to be (*R*).^[11]

Simultaneously, we also performed density functional theory calculations on the reaction highlighted in Scheme 5 using ligand **L2**. We used this ligand as it has provided a firm basis for rationalizing the origin of enantioselectivity of Pd-catalyzed asymmetric allylic alkylation reactions.^[12] Moreover, this ligand provided the same major enantiomer as the optimal ligand **L3** in our allylation process.



Scheme 6. Enantioselective allylic alkylation reactions.



Scheme 7. Stereochemical determination of 8.

We used the B3LYP-D3BJ functional^[13a,b] with the polarizable continuum model^[13c,d] (with the DCM parameters) to describe the solvent. We used a combined basis set of a Stuttgart-Dresden pseudo potential on Pd^[14] and the Pople basis set 6-311G(d,p)^[15] on all other atoms using Gaussian 16.^[16] The full procedure and optimized structures are outlined in the supporting information. The reaction pathway for both stereoisomers proceeds through a pre-reaction complex from which the transition state is reached leading to the corresponding products. The relevant free energies, relative to the pre-reaction complex of the (*R*)-isomer are summarized in Table 1.

Table 1 shows that the pre-reaction complex that leads to the (*R*)-enantiomer is lower in free energy than the corresponding (*S*)-enantiomer by 8.0 kJ mol^{−1}. In addition, the free energy of the transition state that generates the (*R*)-enantiomer is lower than that for (*S*) by 16.4 kJ mol^{−1}. Hence, if we assume that the pre-reaction complex forms under equilibrium conditions, we expect that, at room temperature, 96% will be in a configuration that ultimately leads to (*R*)-3b. The lower transition state barrier for formation of the (*R*)-enantiomer will also lead to a higher rate. Assuming complete equilibration in the pre-reaction complex and standard Eyring behaviour, the rate leading towards (*R*)-3b will be approximately 19 times faster than that for formation of (*S*)-3b. We do note however, that the barriers in both cases are low enough for facile reaction, in line with the experimental observations. Our calculations also allow us to rationalize why the free energy for the pre-reaction complex for the (*R*)-pathway (and its barrier to reaction) are lower than for the (*S*)-pathway. Specifically, as shown in Figure 2, both I, the pre-reaction complex for the (*R*)-pathway, and II, the pre-reaction complex for the (*S*)-pathway, are stabilized by a strong interaction with an N–H group on the catalyst.^[11] However, in case of I, there is also the opportunity for a stabilizing CH/π interaction between the cyclohexyl moiety on L2 with the pendant phenyl group on the lactone with

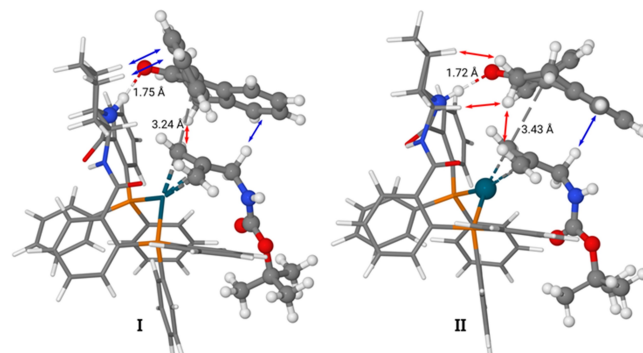


Figure 2. Optimized pre-reaction complex geometries for the reaction of 1 and 2b for the (*R*)-isomer (I) and the (*S*)-isomer (II). Red arrows indicate steric clashes, whereas blue arrows indicate attractive interactions. All distances indicated with red arrows are between 1.9 Å and 2.3 Å. All distances indicated with blue arrows are between 2.6 Å and 3.0 Å. The strong N–H...O hydrogen bond and the forming C–C bond are also indicated in each figure with their corresponding distances.

distances between 2.6 Å and 3.0 Å.^[17] In II these are replaced by steric clashes between the cyclohexyl group on L2 and the aliphatic CH₂ on the ketone as evidenced by the short contacts (1.9 Å–2.3 Å). This will lead to a higher (free) energy for II. Moreover, this difference will be exacerbated in the transition state, leading to the relatively higher transition state for the (*S*)-pathway relative to the (*R*)-pathway, as shown in Table 1.

Having successfully synthesised a series of polycyclic piperidines that contain orthogonal functionality, attention turned toward examining product functionalization. The oxidative cleavage of the *exo*-alkene in 7e was therefore investigated. Boc-protection of the nitrogen followed by oxidative cleavage using RuCl₃ and NaIO₄ in the presence of 2,6-lutidine allowed the formation of the desired ketone as a mixture of carbamate rotamers.^[18] Subsequent deprotection with TFA led to product 13 in 67% yield over three steps (Scheme 8). Notably, compound 13 closely resembles an intermediate converted into haouamine A by Fürstner and co-workers.^[19] The imine functionalization of tetralone-based product 14 (derived from TFA promoted Boc-removal and cyclization of 3b) was also investigated. Using allyldiethylaluminum in CH₂Cl₂ at room temperature, we were able to convert compound 14 to the homoallylic amine product 15 as a single diastereomer (stereochemistry not determined) in a moderate yield.

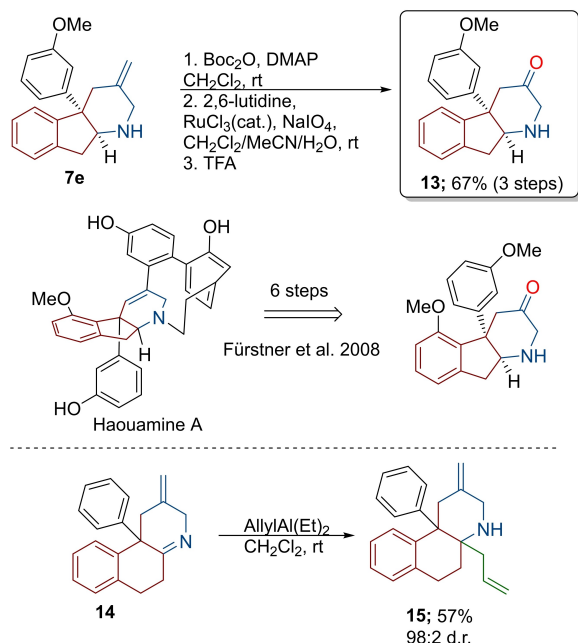
Conclusions

In conclusion, we report that indanone and tetralone-based fused piperidines bearing orthogonal functionality can be readily synthesized and chemoselectively functionalized, providing an efficient and robust methodology to these important nitrogen-containing building blocks. In addition, the key allylic alkylation step can be carried out with useful levels of enantiocontrol when the Trost ligand series is used. Calculations suggest that enantiodiscrimination with ligand L2 stems from a favored stabilizing CH/π interaction between the cyclohexyl

Table 1. Free energies for the formation of the pre-reaction complexes I & II, $\Delta_f(\Delta G)$, and the transition state for the reaction of 1 with 2b, catalyzed by Pd-L2, $\Delta^\ddagger G$, leading to (*R*)/(*S*)-3b.^[a]

	$\Delta_f(\Delta G) / \text{kJ mol}^{-1}$	$\Delta^\ddagger G / \text{kJ mol}^{-1}$
(<i>R</i>)-pathway	0.0	43.7
(<i>S</i>)-pathway	8.0	60.1

[a] All free energies (given in kJ mol^{−1}) are relative to the pre-reaction complex for the (*R*)-enantiomer.



Scheme 8. Representative functionalization reactions.

moiety on the chiral ligand with the pendant phenyl group of the ketone substrate, with the alternative pathway resulting in a destabilizing interaction of the ligand and the cyclic ketone moiety.

Supporting Information

The authors have cited additional references within the Supporting Information (Ref. [21–30]).

Acknowledgements

We are grateful to the EPSRC, the Horizon 2020-MSCA (ITN-EJD CATMEC 14/06-721223) and Government of National Unity Libyan Embassy for funding.

Conflict of Interests

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

Keywords: piperidine • allylic alkylation • catalysis • diastereoselective • enantioselective

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- [11] Deposition numbers 2094703 (for **4a**), and 2323697 (for **12**) contain the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service.
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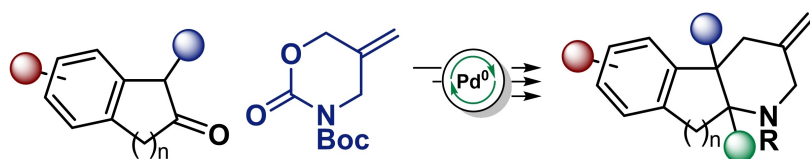
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Manuscript received: January 10, 2024

Accepted manuscript online: February 6, 2024

Version of record online: ■■, ■■

RESEARCH ARTICLE



We report a stereoselective Pd-catalyzed annulation strategy to linearly fused polycyclic piperidines from readily available substrates.

These products can be chemoselectively functionalized to generate analogs that represent common substructures in bioactive compounds.

Dr. L. Hoteite, Dr. B. D. W. Allen, M. E. A. Elhajj, Prof. A. J. H. M. Meijer*, Prof. J. P. A. Harrity*

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A Pd-Catalyzed Annulation Strategy to Linearly Fused Functionalized N-Heterocycles

