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The Construction of Chiral Fused Azabicycles Using a Pd-Catalyzed Allylic Substitution Cascade and Asymmetric Desymmetrization Strategy

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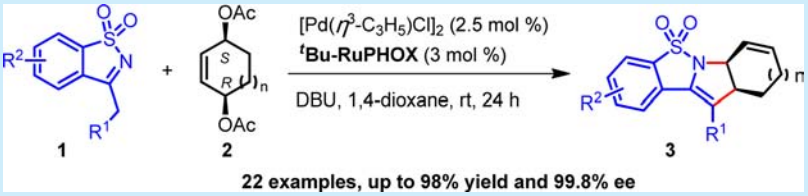
Supporting Information



ABSTRACT: A highly enantioselective Pd-catalyzed asymmetric allylic substitution cascade of cyclic *N*-sulfonylimines with an accompanying asymmetric desymmetrization has been developed for the construction of fused tetrahydroindole derivatives bearing two chiral centers. Mechanistic studies confirmed that the cascade reaction proceeds by initial allylic alkylation and subsequent allylic amination. The first alkylation is a chirality-control step and represents an asymmetric desymmetrization of *cis*-cyclic allyl diacetates. The reaction has been performed on a gram scale, and the desired products can take part in several transformations.

Chiral fused azabicycles represent an important class of compounds because of their prevalence in bioactive natural products and chiral drugs.[1] Among these, azabicycles possessing octahydroindole skeletons are some of the most important.[2] For example, such compounds can serve as key intermediates in the synthesis of angiotensin-converting enzyme inhibitors (ACEIs) such as trandolapril and perindopril.[3] Several strategies for the synthesis of this skeleton have been reported,[4] but these methodologies can only be used for the synthesis of a small variety of azabicyclic products using a limited number of substrates. Therefore, an efficient pathway toward the synthesis of diverse chiral fused azabicyclic octahydroindoles from simple and abundant starting materials remains challenging.

Pd-catalyzed allylic substitution is a powerful synthetic tool for the formation of C−C and C−X bonds (X = N, O, S, etc.).[5] Asymmetric cascade reactions that utilize allylic substitutions provide an efficient pathway for the construction of heterocycles.[6,7] Two strategies utilizing asymmetric cascade reactions based on Pd-catalyzed allylic substitutions have been adopted for the construction of heterocycles. One utilizes a double asymmetric allylic substitution, which allows only the synthesis of single heterocycles bearing a few chiral centers, with somewhat modest catalytic behavior in most cases.[6] A different methodology utilizes an asymmetric conjugate addition−allylic substitution cascade, which also gives single heterocyclic products but with multiple chiral centers and excellent stereoselectivities.[7]

Saccharin-derived cyclic *N*-sulfonylimines are an intriguing class of synthons and have been used in several asymmetric reactions such as hydrogenations,[8] nucleophilic additions,[9] and cycloadditions.[10] *N*-Sulfonylimines have also been used as binucleophiles for the construction of chiral piperidine rings in metal-free asymmetric cascade reactions (sequential C- and N-alkylations) (Scheme 1a).[11] On the basis of our previous work concerning metal-catalyzed asymmetric allylic substitutions[12] and the use of *N*-sulfonylimines as binucleophiles in organocatalyzed asymmetric cascade reactions,[13] we herein report the efficient construction of fused chiral octahydroindole derivatives bearing multiple chiral centers. The strategy employs *N*-sulfonylimines as binucleophiles and *cis*-cyclic allyl diacetates as allylic substrates[14] in a Pd-catalyzed asymmetric allylic substitution cascade with an accompanying asymmetric desymmetrization (Scheme 1b).

Initially, we carried out the Pd-catalyzed asymmetric allylic substitution cascade of cyclic *N*-sulfonylimine **1a** and *cis*-cyclohex-2-ene-1,4-diyl diacetate (**2a**) for screening of the reaction conditions. After examination of several chiral ligands, we found that our previously developed ^{t}Bu-RuPHOX[15] ligand is the best one for the reaction.[16] The effects of different Pd sources, solvents, and bases on the cascade reaction were then examined.[16] The optimal reaction conditions were found to be a catalyst system consisting of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ and ^{t}Bu-RuPHOX in the presence of DBU in 1,4-dioxane under a N_2 atmosphere at 25 °C (Scheme 2).

Next, the applicability of the asymmetric allylic substitution cascade reaction of **1** bearing different R^1 and R^2 substituents

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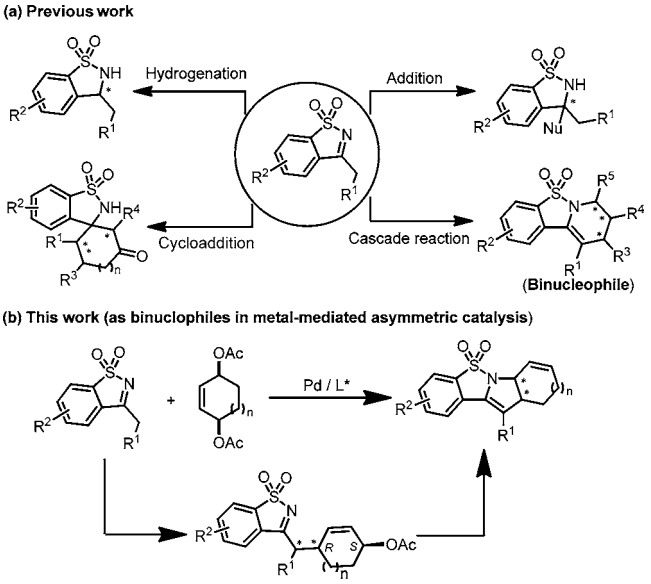
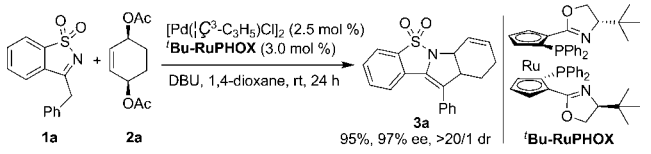
Scheme 1. Use of *N*-Sulfonylimines in Asymmetric Catalysis



Scheme 2. Screening of the Reaction Conditions

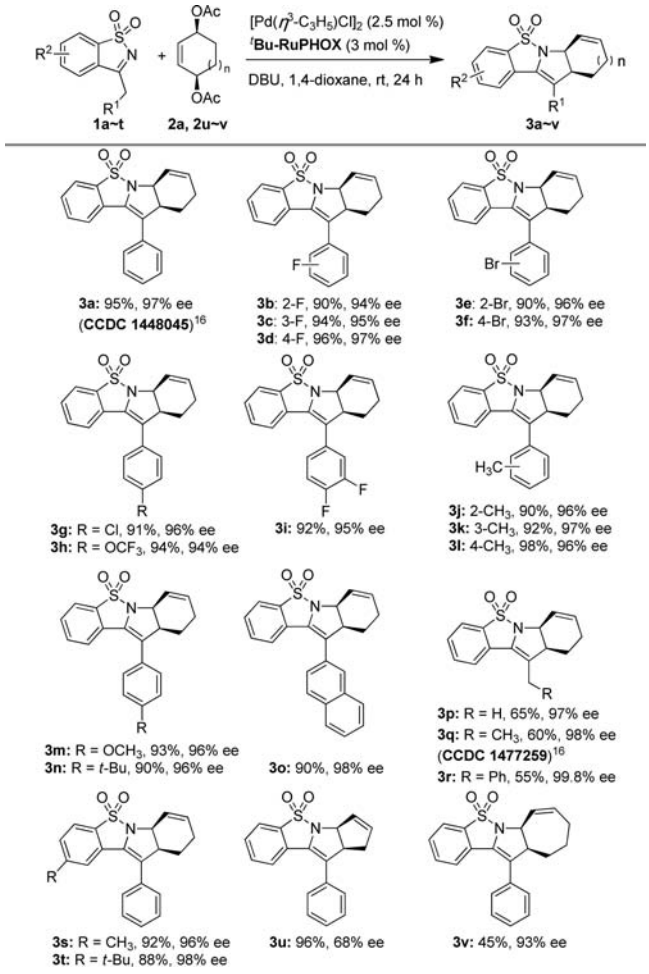


was evaluated (Scheme 3). *N*-Sulfonylimines with an electron-withdrawing group located on the phenyl ring were first investigated. Similar to **1a**, all of these substrates (**1b**–**h**) provided the desired products in high yields with excellent diastereo- and enantioselectivities. We were pleased to discover that the asymmetric catalytic results for the above reactions were unaffected by steric hindrance. The reaction of **1i** containing two F atoms at the 3- and 4-positions of the phenyl ring also gave excellent catalytic results. *N*-Sulfonylimines **1j**–**n** bearing electron-donating substituents were also examined. The desired products were obtained in yields in excess of 90% with excellent enantioselectivities. When the phenyl ring was replaced by a naphthalene (**1o**), excellent catalytic results were also observed. Finally, the aromatic ring of the *N*-sulfonylimines was replaced by aliphatic substituents (**1p**–**r**). To our delight, these substrates all provided excellent enantioselectivities, albeit in moderate yields, with **3r** bearing a Ph group being obtained with 99.8% ee. We also carried out the asymmetric cascade reaction by using a racemic *trans*-cycloalkenediol diacetate instead of *cis*-isomer **2a**.[17] Full conversion but complicated products were obtained even after 72 h.[16]

Substrates **1s** and **1t** with different R^2 substituents (Me and *t*-Bu, respectively) were next examined. High yields and excellent enantioselectivities were still obtained for the cascade reaction. In addition, a high yield but moderate enantioselectivity was obtained when a five-membered cyclic diacetate (**2u**) was used. However, the use of a seven-membered cyclic diacetate (**2v**) gave the corresponding product with high enantioselectivity but low yield.

To evaluate the practicality of this catalytic process, a gram-scale reaction of **1a** with **2a** was carried out under the optimal reaction conditions. As shown in Scheme 4, the desired product

Scheme 3. Substrate Scope[a]



[a]Using the optimal reaction conditions shown in Scheme 2 with ^{t}Bu-RuPHOX as the ligand in 1,4-dioxane. All of the reactions afforded >20:1 dr. Isolated yields are shown. The ee values were determined by HPLC using chiral Daicel columns.

3a was obtained in 92% yield with 94% ee. **3a** has the potential to participate in a variety of transformations. First, the SO_2 group of **3a** and the double bond of the enamide were removed and reduced simultaneously using sodium naphthalide (**4**, pathway a).[10a] The double bond of the cyclohexene ring can also be hydrogenated using $RuCl_2(PPh_3)_3$ as a catalyst under 40 bar H_2 at room temperature (**5**, pathway b). When **3a** is treated with H_2 and a Pd/C catalyst (pathway c), the above two double bonds can be reduced simultaneously to afford octahydroindole **6** in 99% yield with 1:1 dr. Alternatively, the double bond of the enamide can be reduced in the presence of $Et_3SiH/BF_3{\cdot}Et_2O$ to give **7** (pathway d),[13c] which can then be subjected to hydrogenation (pathway c) for 2 h to give octahydroindole **8** in 80% yield with 95% ee after a simple recrystallization. Finally, the double bond of the enamide can be oxidized regioselectively by treating **3a** with *m*-CPBA, affording the oxidized product **9** in high yield and ee; **9** is a key intermediate for the synthesis of chiral alicyclic amino alcohols that possess pharmacological properties.[18]

In order to determine the reaction pathway, the asymmetric allylic substitution cascade of **1a** and **2a** was carried out using the above optimal reaction conditions at −30 °C for 24 h, with the exception that THF was used in place of 1,4-dioxane

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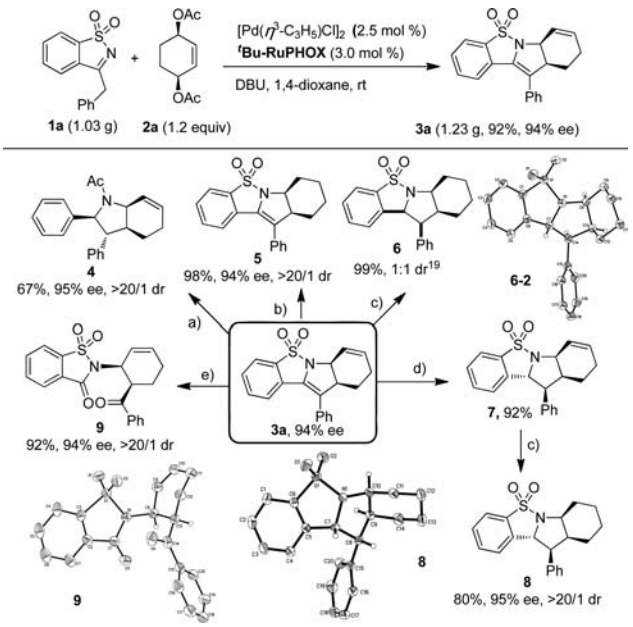
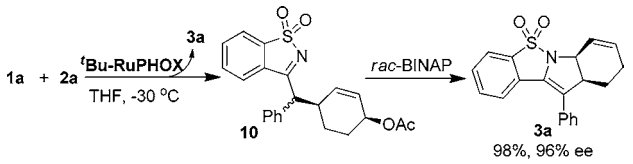
Scheme 4. Large-Scale Synthesis of 3a and Its Transformations[a]



[a]Conditions: (a) Sodium naphthalide, DME, −78 °C to rt, 2 h; Ac_2O, DMAP, DCM, 4 h. (b) $RuCl_2(PPh_3)_3$, K_2CO_3, CH_3OH, 40 bar H_2, 12 h. (c) Pd/C, CH_3OH, H_2 balloon, 12 h. (d) $Et_3SiH/BF_3{\cdot}Et_2O$, DCM, −20 °C, 24 h. (e) *m*-CPBA, DCM, −20 °C, 8 h.

(Scheme 5). A mixture of diastereomers **10** was obtained as the major product with only a trace amount of **3a** and recovery of

Scheme 5. Reaction Pathway

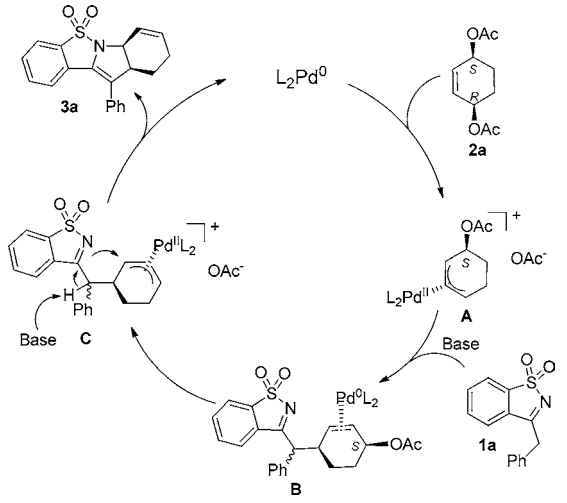


the remaining starting material. Without further isolation, the mixture of **10** was subjected to further transformations using racemic BINAP as a ligand, providing the desired product **3a** in 98% yield with 96% ee.

The above results suggested that the fused azacycle **3a** is constructed via an allylic alkylation followed by an allylic amination. The reaction pathway is envisaged to proceed as follows (Scheme 6). First, combination of *cis*-**2a** with L_2Pd^0 provides allyl-Pd complex **A**. As expected, the process represents an asymmetric desymmetrization of *cis*-**2a** because L_2Pd^0 prefers to attack the *R*-chiral carbon of **2a**. Complex **A** then reacts with nucleophile **1a** to give alkylated intermediate **B**. Finally, **B** takes part in the next allylic amination, giving the terminal fused heterocycle **3a** in high yield and enantioselectivity via allyl-Pd complex **C**. The original catalyst system L_2Pd^0 is subsequently regenerated. It is clear that the asymmetric desymmetrization is the chirality-control step and that the chirality of **3a** is determined by **B**.

In summary, we have developed an efficient pathway for the construction of chiral fused azabicycles using a Pd-catalyzed allylic substitution cascade and asymmetric desymmetrization strategy. Under the optimal reaction conditions, a series of cyclic *N*-sulfonylimines can be used to give the desired products

Scheme 6. Proposed Reaction Mechanism



in high yields with up to 99.8% ee. The reaction was performed on a gram scale, and the corresponding products allowed for several transformations. Mechanistic studies confirmed that the cascade reaction undergoes an allylic alkylation followed by an allylic amination. The first alkylation is the chirality-control step and represents an asymmetric desymmetrization of *cis*-**2**. The alkylated intermediates determine the configuration of the terminal cascade products **3** via a subsequent amination step.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b03529.

Crystallographic data for **3a** (CIF)
Crystallographic data for **3q** (CIF)
Crystallographic data for **6-2** (CIF)
Crystallographic data for **8** (CIF)
Crystallographic data for **9** (CIF)
Experimental procedures and characterization data for all reactions and products, including ^1H and ^{13}C NMR spectra, HPLC data, and crystal data (PDF)

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Notes

The authors declare no competing financial interest.

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