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Construction of Chiral 1,3-Diamines through Rhodium-Catalyzed Asymmetric Arylation of Cyclic *N*-Sulfonyl Imines

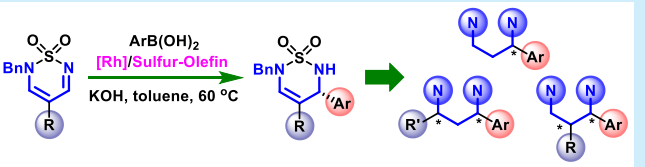
Chun-Yan Wu[†] and Ming-Hua Xu*[,†,‡]

[†]State Key Laboratory of Drug Research, Shanghai Institute of Materia Medica, Chinese Academy of Sciences, 555 Zu-chongzhi Road, Shanghai 201203, China

[‡]Shenzhen Grubbs Institute and Department of Chemistry, Southern University of Science and Technology, 1088 Xueyuan Boulevard, Shenzhen 518055, China

Supporting Information

ABSTRACT: A rhodium/sulfur−olefin complex catalyzed asymmetric 1,2-addition of arylboronic acids to six-membered 1,2,6-thiadiazinane 1,1-dioxide-type cyclic imines to access highly optically active sulfamides (95−99% ee) has been developed. By taking advantage of the simple functional group transformations, an interesting array of valuable chiral 1,3-diamines with different substitution patterns can be readily obtained in a highly enantioenriched manner.



Chiral 1,3-diamines are common motifs present in a large number of organic molecules including natural products,[1] bioactive compounds,[2] pharmaceutical agents,[3] as well as chiral ligands and catalysts[4] (Figure 1). Due to this great importance,

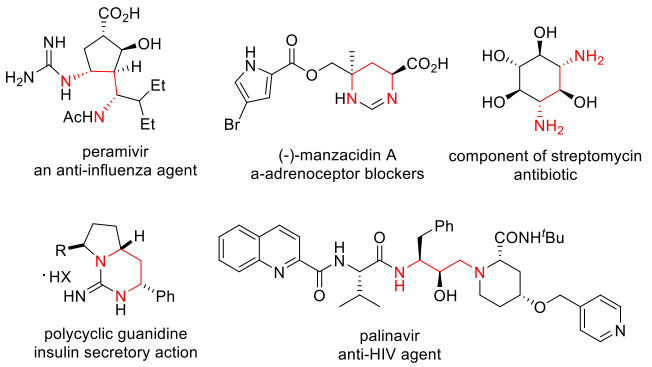


Figure 1. Examples of chiral 1,3-diamine-based bioactive compounds.

the construction of chiral 1,3-diamine scaffolds is known as an important subject in organic synthesis. Accordingly, various synthetic methods to access molecules containing a chiral 1,3-diamine framework have been developed. Asymmetric Mannich-type reaction using a certain type of nucleophiles such as nitriles or enamides can lead to 1,3-dinitrogen-containing products, but the generation of 1,3-diamines often requires further reduction with hydride reagents.[5] The stereoselective ring-opening reaction of the substituted aziridines with nucleophiles could also provide highly enantioenriched 1,3-diamines efficiently.[6] Additionally, chiral 1,3-diamines can be prepared by asymmetric conjugate addition reaction[7] and enantioselective 1,3-dipolar cycloaddition reaction[8] followed by ring cleavage. Notably, transition-metal-catalyzed allylic amination and intramolecular diastereoselective C−H amination offered new strategies for the synthesis of optically active 1,3-diamines.[9] However, most of the available methods usually involve lengthy processes, thus it still remains desirable to develop concise and highly stereoselective approaches capable of accessing structurally diverse and synthetically versatile chiral 1,3-diamines.

Rhodium-catalyzed asymmetric addition of organoboron reagents to imines has proved to be a powerful strategy for the straightforward synthesis of diverse chiral amines. Recently, numbers of cyclic imines were employed in rhodium-catalyzed enantioselective addition of organoboron reagents, allowing for access to a broad range of highly optically active cyclic amine compounds containing multifunctional groups.[10,11] Among them, we have succeeded in efficient asymmetric addition to 1,2,5-thiadiazolidine 1,1-dioxide-type cyclic ketimines using a simple chiral phosphite−olefin as a ligand.[11f,k] These methods are useful for the synthesis of highly enantioenriched 1,2-diamines through ring cleavage. In considering the construction of a 1,3-diamine framework, we envisaged that the incorporation of one more carbon in the heterocyclic system, namely, employing 1,2,6-thiadiazinane 1,1-dioxide-type cyclic imines as substrates for Rh-catalyzed asymmetric addition, would be a potential protocol for accessing the 1,3-diamine skeleton. Herein, we describe our successful development of such an approach that could provide facile access to highly enantioenriched diversely substituted chiral cyclic sulfamides bearing 1,3-diamine scaffolds through the use of a simple sulfur−olefin ligand (Scheme 1). After further synthetic transformations, versatile acyclic and polycyclic chiral 1,3-diamines were easily attained.

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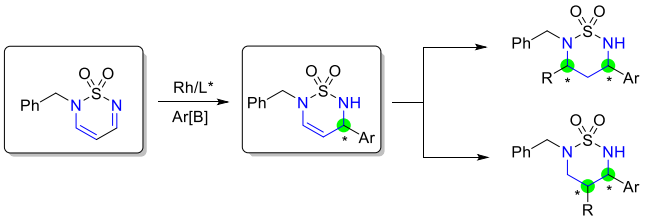
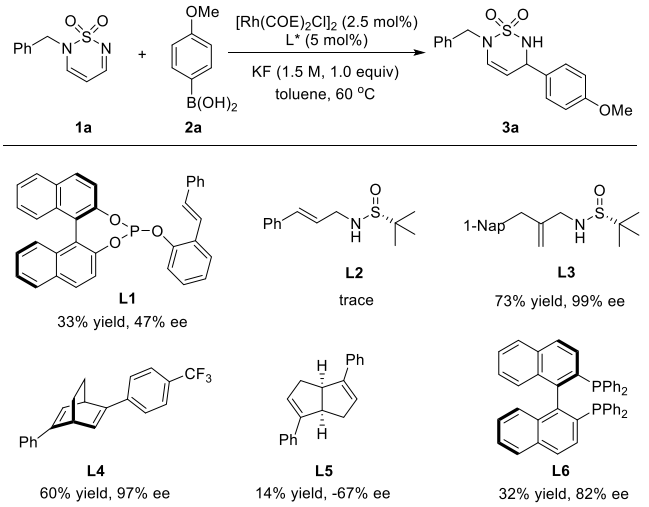
Scheme 1. Addition Strategy to Chiral 1,3-Diamines



The six-membered 1,2,6-thiadiazinane 1,1-dioxide-type cyclic imine substrates could be readily prepared by reaction of sulfuric diamide with 1,1,3,3-tetramethoxypropane or 1,3-dicarbonyl compounds through hydrogen chloride-promoted dehydration.[12] We began our work by evaluating the reaction between **1a** and *p*-methoxyphenylboronic acid **2a** in KF (1.5 M)/toluene at 60 °C, in the presence of 2.5 mol % of $[Rh(COE)_2Cl]_2$ and 5 mol % of various representative chiral olefin ligands (**L1**−**L5**) that are prepared in our laboratory (Scheme 2). Surprisingly, the use of the Rh/phosphite−olefin

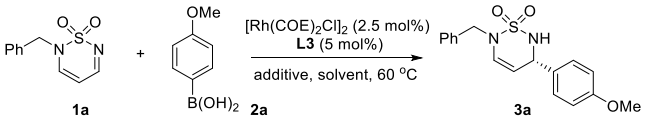
Scheme 2. Ligand Screening



(**L1**) catalyst system that works well for the previous five-membered 1,2,5-thiadiazolidine 1,1-dioxide-type ketimines[11k] could only deliver the desired product **3a** in low yield (33%) and poor enantioselectivity (47% ee). It was exciting to notice that the branched sulfur−olefin ligand **L3** exhibited excellent catalytic reactivity and stereocontrol for 1,2-addition, giving nearly optically pure cyclic sulfamide **3a** with commendable yield (73% yield, 99% ee). C_1-symmetric chiral diene **L4** also displayed good reactivity and great enantioselectivity in the reaction (60% yield, 97% ee). Other ligands including BINAP can also produce the desired adduct, although the yields and ee's were inferior (**L5**, **L6**).

Encouraged by the promising result obtained with branched chiral sulfur−olefin ligand **L3**, further investigations on additives, solvents, and catalyst loading were carefully conducted (Table 1). Among the various additives screened (entries 1−5), KOH was found to be the best and gave the highest yield (86%) with maintaining enantioselectivity (99% ee) (entry 5). In the presence of 1.5 equiv of aqueous KOH (1.5 M), the reaction afforded **3a** in a slightly increased yield (88%, entry 6). Solvent screening showed that toluene was the best choice (entries 6−8). Slightly declined reaction yield was

Table 1. Optimization of Reaction Conditions[a]



entry	solvent	additive[b]	yield (%)[c]	ee (%)[d]
1	toluene	KF (1.0 equiv)	73	99
2	toluene	K_3PO_4 (1.0 equiv)	77	98
3	toluene	KHF_2 (1.0 equiv)	64	99
4	toluene	NEt_3 (1.0 equiv)	57	99
5	toluene	KOH (1.0 equiv)	86	99
6	toluene	KOH (1.5 equiv)	88	99
7	dioxane	KOH (1.5 equiv)	44	98
8	DCE	KOH (1.5 equiv)	trace	-
9[e]	toluene	KOH (1.5 equiv)	82	99
10[g]	toluene	KOH (1.5 equiv)	78	99
11[g]	toluene	KOH (1.5 equiv)	73	99

[a]The reaction was carried out with 0.2 mmol of cyclic *N*-sulfonyl aldimine **1a** and 2.0 equiv of *p*-methoxyphenylboronic acid **2a** in the presence of 5 mol % of [Rh]/**L3** with 2.0 mL of solvent at 60 °C for 8 h. [b]Unless noted, 1.5 M solution was used. [c]Isolated yield. [d]Determined by Chiral HPLC. [e]The reaction was carried out in the presence of 3 mol % of [Rh]/**L3**. [f]With 1.5 equiv of *p*-methoxyphenylboronic acid **2a**. [g]With 1.2 equiv of *p*-methoxyphenylboronic acid **2a**.

observed when we tried to lower the catalyst loading to 3 mol % (entry 9). When the reaction was carried out with the use of less amount of arylboronic acid, a decrease of the yield was observed while maintaining excellent enantioselectivity (entries 10 and 11).

With the optimal conditions in hand, we next investigated the substrate scope of this asymmetric arylation (Scheme 3). As revealed in Scheme 3, we were pleased to find that the Rh/sulfur−olefin (**L3**) catalyst system could nicely apply to various arylboronic acids with either electron-donating or -withdrawing group(s) on the phenyl ring at different positions, allowing the formation of the corresponding 3-aryl-substituted sulfamide products with extremely high enantioselectivities (98−99% ee, **3a**−**3l**). Sterically bulky boronic acids can also provide the desired 1,2-adducts smoothly (**3l**, **3m**). Notably, the reaction of heterocyclic boron reagents also proceeded well. With 3-thienylboronic acid and 3-furanboronic acid, the addition products were obtained with extremely high enantioselectivity (98% ee) and medium yield (**3o**, **3p**). Moreover, substrates bearing various functional groups such as halogen, vinyl, and phenyl at the 4-position were also suitable for this catalytic system, providing the corresponding products (**3q**−**3t**) in satisfactory yields with uniformly high enantioselectivities. To further extend the substrate scope, we also investigated the cyclic imines with other alkyl groups on the nitrogen. To our delight, when substrates having *N*-methyl and *N*-PMB groups were employed, the corresponding products (**3u**, **3v**) were obtained in 68% and 98% yields with no change of the enantioselectivity (99% ee). It is noteworthy that the reaction at 1 mmol scale with **1a** and **2b** proved also successful, affording **3b** in nearly quantitative yield with no notable loss of enantioselectivity (95% yield, 99% ee).

To showcase the synthetic utility of this protocol, we next carried out a series of experiments for product transformation to construct structurally diverse chiral 1,3-diamines (Scheme 4). By taking advantage of the enamide functionality, allylation

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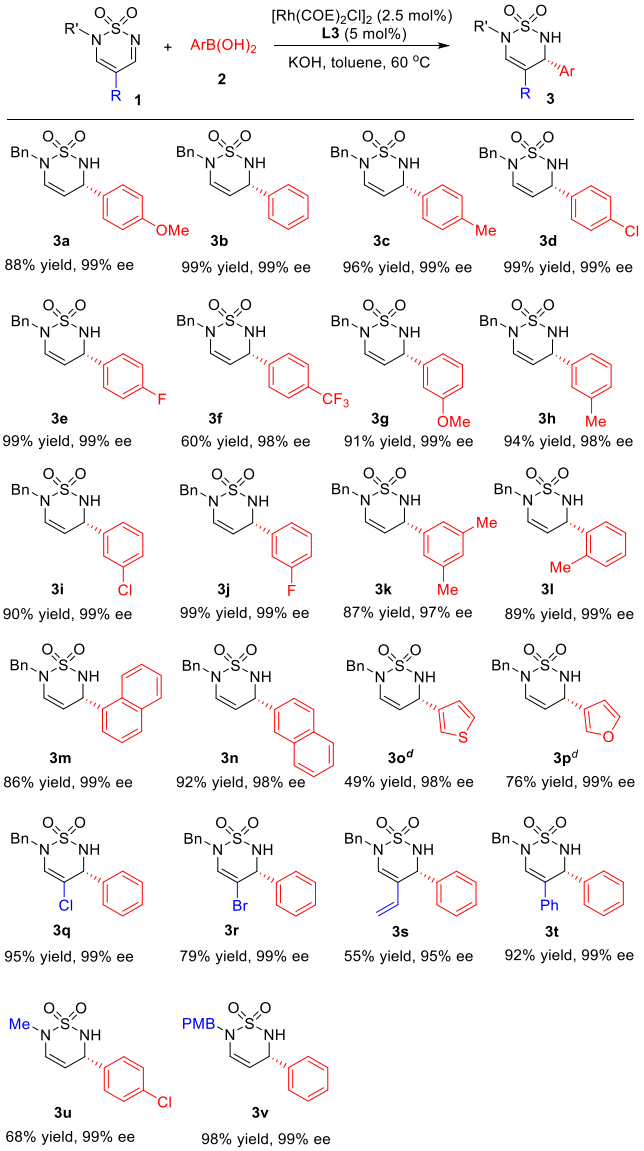
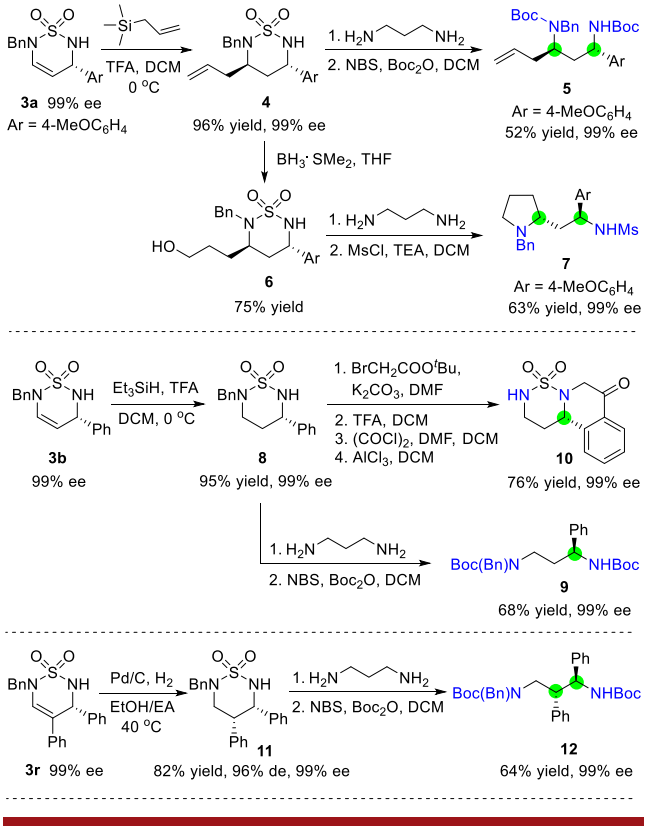
Scheme 3. Rh-Catalyzed Asymmetric Addition of Cyclic *N*-Sulfonyl Imines 1 with Arylboronic Acids[a,b,c]



[a]The reaction was carried out with 0.2 mmol of cyclic imine **1**, 2.0 equiv of arylboronic acid **2** in the presence of 5.0 mol % of [Rh]/**L3**, and KOH (1.5 M, 1.5 equiv) in 2.0 mL of toluene at 60 °C for 2~12 h. [b]Isolated yield. [c]Determined by Chiral HPLC. [d]4.0 equiv of arylboronic acid was used.

of **3a** with allyltrimethylsilane successfully proceeded in the presence of TFA, furnishing the sole 3,5-*anti*-substituted 1,2,6-thiadiazinane 1,1-dioxide **4** in excellent yield in a highly enantiomerically enriched manner (99% ee).[13] The structure of **4** was confirmed by X-ray diffraction analysis, and the absolute configurations of two carbon stereogenic centers were determined to be (3*R*, 5*S*) (Figure 2). Assuming an analogous addition mechanism, the same stereochemistry of the obtained products **3** could be assigned. Ring cleavage of the cyclic sulfamide **4** with trimethylenediamine under reflux conditions,[9b] followed by the *N*-Boc-protection, could lead to acyclic chiral 1,3-disubstituted 1,3-diamine **5** bearing useful allyl and aryl functional groups without any loss of enantiopurity (99% ee). On the other hand, hydroboration of the terminal alkenyl of cyclic sulfamide **4** gave alcohol intermediate **6**. Exposure of

Scheme 4. Synthesis of Chiral 1,3-Diamines



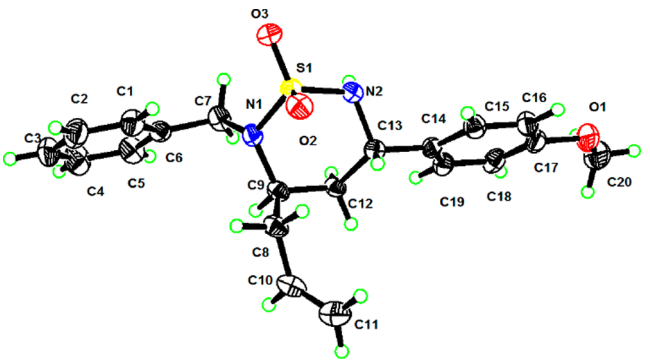


Figure 2. X-ray crystal structure of the cyclic sulfamide **4**.

6 under ring-opening conditions followed by the treatment with MsCl/TEA led to a sequential cyclization to afford a pyrrolidine-based chiral 1,3-diamine **7**, which is otherwise difficult to prepare. In the other case, triethylsilane reduction of adduct **3b** under trifluoroacetic acid conditions gave the cyclic sulfamide product **8** without losing enantioselectivity. Accordingly, a simple 1-substituted 1,3-diamine **9** could be attained after ring cleavage. Derivatization of the sulfamide **8** to form a polycyclic compound **10** has also been achieved by N-acetylation and subsequent Friedel–Crafts reaction. In another experiment, hydrogenation of 4-phenyl-substituted adduct **3r** over Pd/C in EA/ethanol at 40 °C was performed. Interestingly, it was found that the reaction proceeded with excellent diastereoselectivity (96% de) and gave *syn*-4,5-substituted product **11** with high enantioselectivity (99% ee). Removal of sulfonyl could then provide enantioenriched 1,2-disubstituted 1,3-diamine **12**. Thus, we were able to flexibly construct an interesting array of 1-substituted, 1,2-disubsti-

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tuted, and 1,3-disubstituted chiral 1,3-diamines which should be useful in organic synthesis and medicinal chemistry.

To summarize, we have developed a highly effective rhodium/sulfur−olefin ligand catalytic system for asymmetric addition of arylboronic acids to six-membered 1,2,6-thiadiazinane 1,1-dioxide-type cyclic imines bearing an enamide moiety. The reaction facilitates the preparation of diverse cyclic sulfamides in high yields with excellent enantioselectivities (95−99% ee) under mild conditions. More importantly, a series of valuable chiral 1,3-diamines with different substitution patterns can be readily obtained in a highly enantioenriched manner by simple conversions. Given the great importance of chiral 1,3-diamines, we believe that this protocol would find applications in related drug discovery studies and asymmetric organic synthesis.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b01633.

Experimental procedures and spectroscopic data of all new compounds (PDF)

Accession Codes

CCDC 1914877 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: xumh@simm.ac.cn or xumh@sustech.edu.cn.

ORCID

Ming-Hua Xu: 0000-0002-1692-2718

Notes

The authors declare no competing financial interest.

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