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Enantioselective Access to Chiral 2-Substituted 2,3-Dihydrobenzo[1,4]dioxane Derivatives through Rh-Catalyzed Asymmetric Hydrogenation

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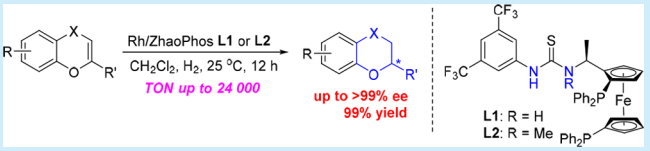
Supporting Information

ABSTRACT: Rh-catalyzed asymmetric hydrogenation of various benzo[*b*][1,4]dioxine derivatives was successfully developed to prepare chiral 2-substituted 2,3-dihydrobenzo-[1,4]dioxane derivatives using ZhaoPhos and *N*-methylation of ZhaoPhos ligands with high yields and excellent enantioselectivities (up to 99% yield, >99% enantiomeric excess (ee), turnover number (TON) = 24 000). Moreover, this asymmetric hydrogenation methodology, as the key step with up to 10 000 TON, was successfully applied to develop highly efficient synthetic routes for the construction of some important biologically active molecules, such as MKC-242, WB4101, BSF-190555, and (*R*)-doxazosin·HCl.



Chiral 2-substituted 2,3-dihydro-1,4-benzodioxane motifs have wide application in a variety of bioactive natural products and therapeutic agents with important biological activities (see Figure 1),[1−10] such as the antihypertensive drug (*R*)-doxazosin,[3] selective α_{2C} adrenergic receptor antagonist,[4] the antidepressant MKC-242,[5] the potent α_{1D}-adrenergic antagonist WB4101,[6] the antihypertensive agent IDR-16084,[7] the 5-HT_{1A} receptor agonist BSF-190555,[8] the flesinoxan hydrochloride,[9] and the anticonvulsant JNJ-26489112.[10]

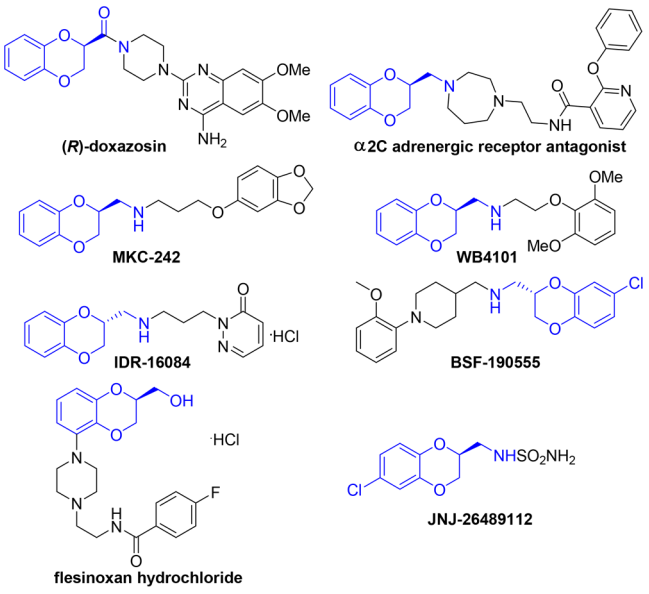


Figure 1. Examples of biologically active molecules containing the chiral 2,3-dihydro-1,4-benzodioxane framework.

In the past decades, several approaches were developed to construct the important chiral 2,3-dihydro-1,4-benzodioxane frameworks. Among them, the enzymatic resolution of racemic 1,4-benzodioxan-2-carboxylic acids and derivatives is a common method.[11,2a] Other methods involved the chiral pool[12] and diastereomeric crystallization with chiral amines.[13] Buchwald and co-workers prepared chiral 1,4-benzodioxanes and derivatives through Pd-catalyzed intramolecular etherification of aryl halides with a chiral alcohol group.[14] Cai and co-workers developed Pd-catalyzed asymmetric intramolecular *O*-arylation coupling reaction via asymmetric desymmetrization to obtain chiral 1,4-benzodioxanes with moderate to excellent enantioselectivities.[15] Zhang and co-workers realized Ir-catalyzed asymmetric hydrogenation of 2-substituted 1,4-benzodioxines to prepare chiral 1,4-benzodioxanes.[16] Because of the great importance of the chiral 1,4-benzodioxane unit, it is necessary to develop a highly reactive and enantioselective catalytic system to construct chiral 1,4-benzodioxanes and derivatives. Transition-metal-catalyzed asymmetric hydrogenation of prochiral unsaturated compounds has been considered to be a direct and atom-

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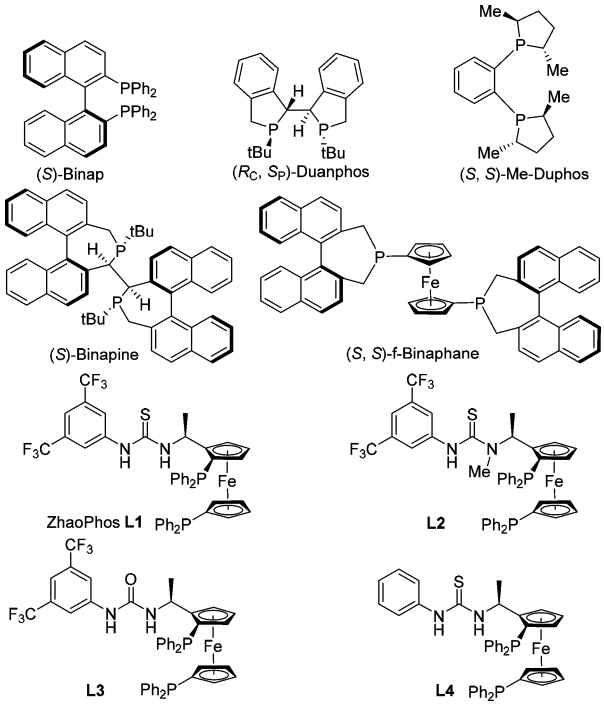
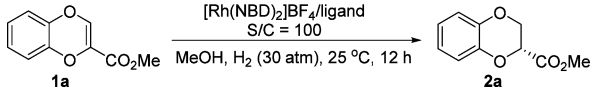


Figure 2. Chiral biphosphine ligands for the Rh-catalyzed asymmetric hydrogenation of methyl benzo[*b*][1,4]dioxine-2-carboxylate **1a**.

Table 1. Screening Ligands for the Rh-Catalyzed Asymmetric Hydrogenation of Methyl Benzo[*b*][1,4]dioxine-2-carboxylate 1a[a]



entry	ligand	conversion[b] (%)	ee[c] (%)
1	(*S*)-Binap	15	55
2	(R_C,S_P)-Duanphos	20	62
3	(*S*,*S*)-Me-Duphos	98	70
4	(*S*)-Binapine	65	77
5	(*S*,*S*)-f-Binaphane	67	84
6	ZhaoPhos **L1**	>99	98
7	**L2**	>99	98
8	**L3**	97	50
9	**L4**	99	96

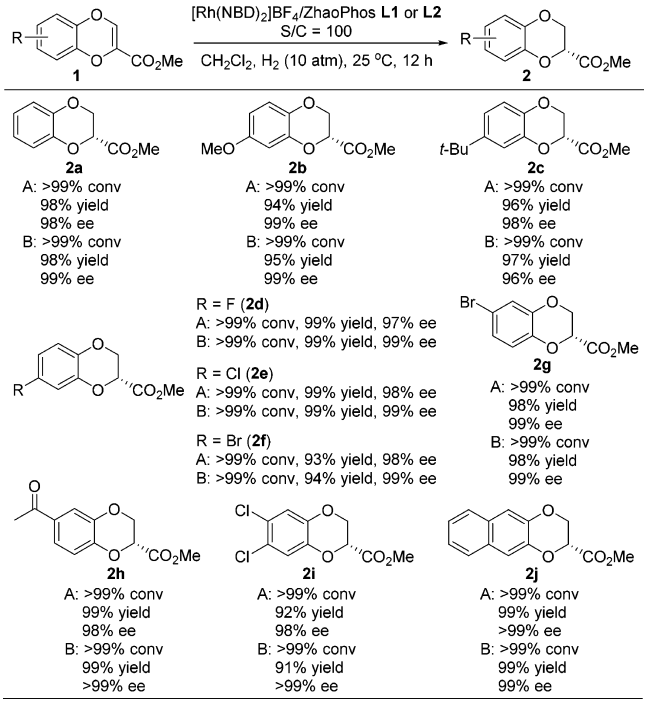
[a]Unless otherwise mentioned, all reactions were carried out with a [Rh(NBD)$_2$]BF$_4$/ligand/substrate ratio of 1:1.1:100 in MeOH, 0.1 mmol substrate **1a**, 30 atm hydrogen, 25 °C, 12 h. [b]Determined by ^{1}H NMR analysis. [c]Determined by HPLC on a chiral phase.

economic synthetic methodology in asymmetric synthesis.[17] Previously, we have successfully applied a bifunctional bisphosphine−thiourea ligand (ZhaoPhos) into Rh-catalyzed asymmetric hydrogenation through the important interactions between the thiourea group and the functional group of substrates with excellent results.[18] On the basis of this theory, we herein successfully realized Rh/ZhaoPhos and *N*-methylation of ZhaoPhos-catalyzed asymmetric hydrogenation of 2-substituted benzo[*b*][1,4]dioxine derivatives with up to 99% yield, >99% enantiomeric excess (ee), and a turnover number (TON) of 24 000.

We began the initial investigation with Rh-catalyzed asymmetric hydrogenation of methyl benzo[*b*][1,4]dioxine-2-carboxylate **1a** as a model substrate to optimize the reaction conditions. Some important bisphosphine ligands such as (*S*)-Binap, (R_C,S_P)-Duanphos, (*S*,*S*)-Me-Duphos, (*S*)-Binapine, (*S*,*S*)-f-Binaphane, and ZhaoPhos (Figure 2) were evaluated in the presence of 1.0 mol % of catalyst under 30 atm H_2 in MeOH at room temperature for 12 h. When ZhaoPhos **L1** was employed, the product **2a** was obtained with full conversion and 98% ee (Table 1, entry 6). Other chiral bisphosphine ligands provided poor to moderate reaction results (15%−98% conversions, 55%−84% ee; Table 1, entries 1−5). Other chiral bisphosphine-(thio)urea ligands (**L2**−**L4**) were also investigated in this transformation. Except for ligand **L3**, which changed from a thiourea motif to urea motif and displayed poor enantioselectivity (50% ee; Table 1, entry 8), others showed satisfactory results (96%−98% ee; Table 1, entries 7 and 9). To our surprise, the *N*-methylation of ZhaoPhos **L2** displayed excellent enantioselectivity, which is the same with ZhaoPhos **L1** (98% ee; Table 1, entry 7); it is possible that one hydrogen bond between the substrate and ligand displayed nearly the same effect with two hydrogen bonds in this asymmetric reaction. The solvent effect was then inspected; the results are summarized in Table S1 in the Supporting Information, and CH_2Cl_2 was selected as the best solvent.

With the optimized reaction conditions in hand, we started to explore the substrate scope of this reaction catalyzed by [Rh(NBD)$_2$]BF$_4$/**L1** and **L2** in CH_2Cl_2. As shown in Scheme 1, a series of methyl benzo[*b*][1,4]dioxine-2-carboxylates (**1a**−

Scheme 1. Scope Study for the Rh-Catalyzed Asymmetric Hydrogenation of Methyl Benzo[*b*][1,4]dioxine-2-carboxylates[a]



[a]Condition A: 0.1 mmol substrate **1**, substrate/ [Rh(NBD)$_2$]BF$_4$/ZhaoPhos **L1** = 1/0.01/0.011 at 25 °C under 10 atm H_2 in 1.0 mL CH_2Cl_2 for 12 h. Condition B: 0.1 mmol substrate **1**, substrate/ [Rh(NBD)$_2$]BF$_4$/**L2** = 1/0.01/0.011 at 25 °C under 10 atm H_2 in 1.0 mL CH_2Cl_2 for 12 h. Conversion was determined by ^{1}H NMR analysis. Isolated yields. The ee value was determined by HPLC on a chiral column.

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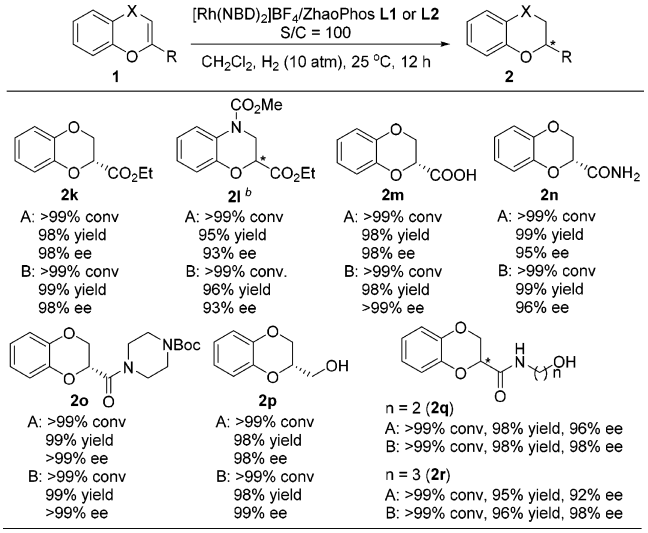
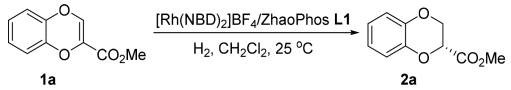
Scheme 2. Scope Study for the Rh-Catalyzed Asymmetric Hydrogenation of 2-Substituted Benzo[*b*][1,4]dioxins[a]



[a]Condition A: 0.1 mmol substrate **1**, substrate/ $[Rh(NBD)_2]BF_4$/ZhaoPhos **L1** = 1/0.01/0.011 at 25 °C under 10 atm H_2 in 1.0 mL CH_2Cl_2 for 12 h. Condition B: 0.1 mmol substrate **1**, substrate/ $[Rh(NBD)_2]BF_4$/**L2** = 1/0.01/0.011 at 25 °C under 10 atm H_2 in 1.0 mL CH_2Cl_2 for 12 h. Conversion was determined by ^1H NMR analysis. Isolated yields. The ee values were determined by HPLC on a chiral column. [b]Reaction temperature = 60 °C.

Table 2. TON Experiments of Rh-Catalyzed Asymmetric Hydrogenation of Methyl Benzo[*b*][1,4]dioxine-2-carboxylate 1a[a]



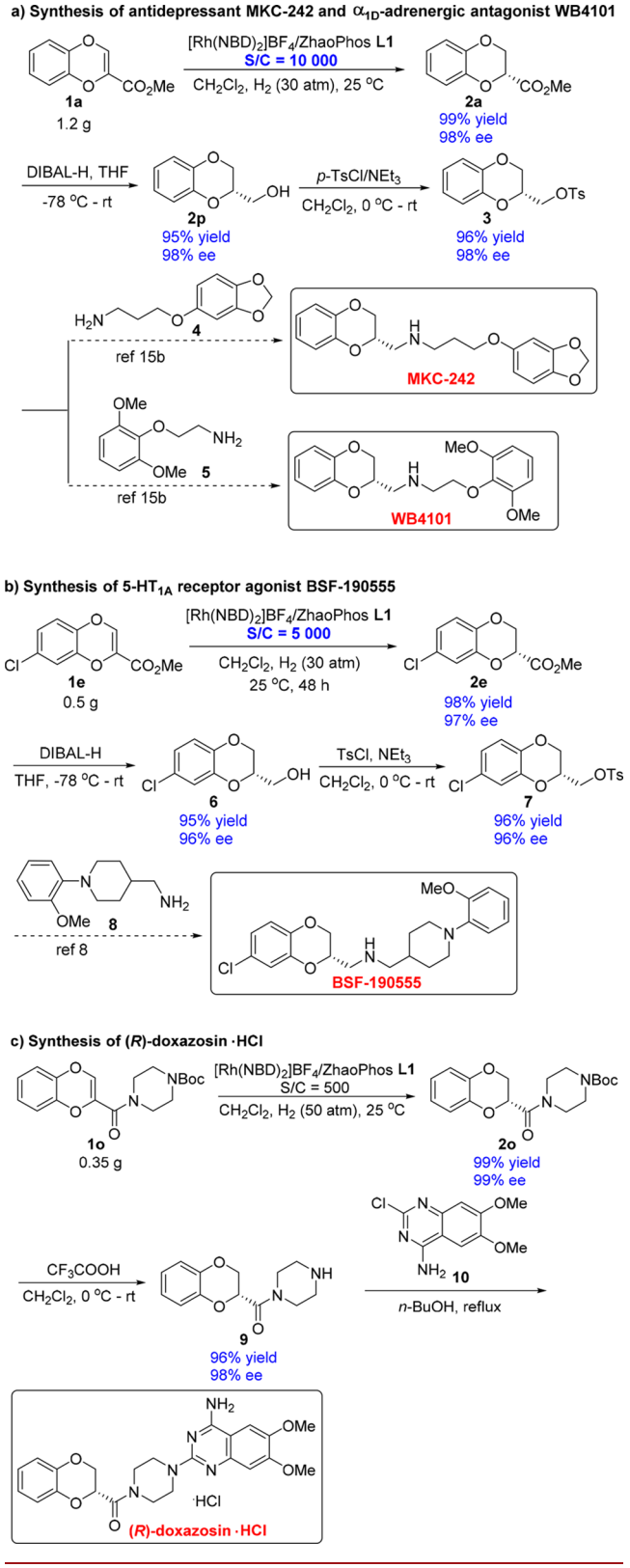
entry	S/C	H_2 (atm)	time (h)	conversion[b] (%)	yield[c] (%)	ee[d] (%)
1	2000	30	36	>99	98	98
2	5000	30	48	>99	98	98
3	10 000	30	52	>99	99	98
4[e]	24 000	60	72	>99	99	97
5[e,f]	24 000	60	72	>99	99	98

[a]Unless otherwise noted, all reactions were performed with a $[Rh(NBD)_2]BF_4$/ZhaoPhos **L1** = 1:1.1 in CH_2Cl_2. [b]Determined by ^1H NMR analysis. [c]Isolated yield. [d]Determined by HPLC analysis using a chiral stationary phase. [e]Reaction temperature = 40 °C. [f]Using ligand **L2**.

1i)[2b,19a] with various substituents on the phenyl ring were hydrogenated smoothly and were catalyzed by both $[Rh(NBD)_2]BF_4$/**L1** and $[Rh(NBD)_2]BF_4$/**L2** with excellent results. The methyl benzo[*b*][1,4]dioxine-2-carboxylates bearing electron-donating groups (**1b** and **1c**) or electron-withdrawing groups (**1d**−**1i**) on the phenyl ring performed well with full conversions, 91%−99% yields, and 96%−>99% ee. The substrate with a sterically hindered naphthyl group **1j** is also converted to the hydrogenation product **2j** with excellent results (99% yield, 99%−>99% ee).

In addition, in order to further investigate the tolerance of substrate scope of this catalytic system, a series of different 2-substituted benzo[*b*][1,4]dioxine derivatives **1k**−**1r**, including 2-carboxyl, 2-amido, and 2-hydroxymethyl group were applied in this asymmetric hydrogenation catalyzed by $[Rh(NBD)_2]$-BF_4/**L1** and **L2**. As shown in Scheme 2, these different 2-substituted benzo[*b*][1,4]dioxins proceeded smoothly to afford the hydrogenation products **2k**−**2r** with excellent results (95%−99% yields, 92%−>99% ee). It was worth noting that the substrate benzo[*b*][1,4]dioxin-2-ylmethanol **1p** reacted well to

Scheme 3. Synthetic Transformations of Hydrogenation Products



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produce compound **2p** with high yield and excellent enantioselectivity, which is the key intermediate for the synthesis of some important biologically active molecules, such as MKC-242 and WB4101.

Our Rh/ZhaoPhos **L1** and *N*-methylation of ZhaoPhos **L2** catalytic system are very highly efficient in this asymmetric hydrogenation. The model substrate methyl benzo[*b*][1,4]-dioxine-2-carboxylate **1a** was hydrogenated well, catalyzed by Rh/ZhaoPhos **L1** with 0.05 mol % (S/C = 2000) catalyst, 0.02 mol % (S/C = 5000), or even with 0.01 mol % (S/C = 10 000). Full conversion, high yield, and 98% ee can be obtained (Table 2, entries 1−3). 99% yield and 97% ee can be obtained when S/C is 24 000 (Table 2, entry 4). The Rh/*N*-methylation of ZhaoPhos **L2** catalytic system also worked well; almost the same excellent results can be afforded with S/C = 24 000 (Table 2, entry 5).

This highly efficient asymmetric hydrogenation methodology demonstrated powerful synthetic utility. As shown in Scheme 3, several great and creative synthetic routes were described to prepare some important biologically active compounds. Hydrogenation product **2a** is the key intermediate for the construction of antidepressant MKC-242 and potent α_{1D}-adrenergic antagonist WB4101, which can be easily obtained through our asymmetric catalytic hydrogenation system in 1.2 g scale with 0.01 mol % catalyst (S/C = 10 000, 99% yield, 98% ee; Scheme 3a). Compound **2a** was reduced by diisobutylaluminum hydride (DIBAL-H) to provide compound **2p**, which was treated with *p*-TsCl (*p*-toluene sulfonyl chloride) to afford compound **3** in 96% yield and without a loss of ee. Finally, compound **3** can react with amines **4** and **5** to give the corresponding MKC-242 and WB4101.[15b] Compound **7** can be easily synthesized through a similar approach, which could be worked as the important intermediate for the construction of 5-HT_{1A} receptor agonist BSF-190555 (Scheme 3b).[8] Compound **2o** was easily obtained in 99% yield and 99% ee through this asymmetric hydrogenation. It then underwent deprotection to give intermediate **9** in 96% yield and 98% ee, which reacted with the amine **10** to afford (*R*)-doxazosin·HCl (Scheme 3c).[12a,19b]

In summary, we successfully developed a Rh/ZhaoPhos and *N*-methylation of ZhaoPhos-catalyzed asymmetric hydrogenation of various benzo[*b*][1,4]dioxine derivatives, a wide range of chiral 2,3-dihydrobenzo[1,4]dioxane derivatives was generated with high yields and excellent enantioselectivities (up to 99% yield and >99% ee, TON up to 24 000). Moreover, this highly efficient asymmetric hydrogenation methodology exhibited powerful synthetic utility. Highly efficient synthetic routes for the construction of important biologically active molecules MKC-242, WB4101, BSF-190555, and (*R*)-doxazosin·HCl were described through this asymmetric hydrogenation methodology with up to 10 000 TON.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b01469.

General remarks; general procedures for the synthesis of **1a−1j**; general procedures for the prpearation of **1k−1s**; characterization of the **1** substrates; screening solvents for the Rh-catalyzed asymmetric hydrogenation of **1a**; general procedure for the asymmetric hydrogenation of **1**; general procedure regarding the high TON experiment of **1a**; NMR and HPLC spectra (PDF)

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Notes

The authors declare no competing financial interest.

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