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Asymmetric [3 + 2] Cycloaddition Employing *N,N′*-Cyclic Azomethine Imines Catalyzed by Chiral-at-Metal Rhodium Complex

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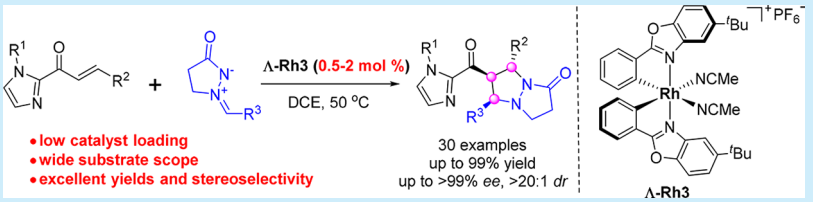
Supporting Information

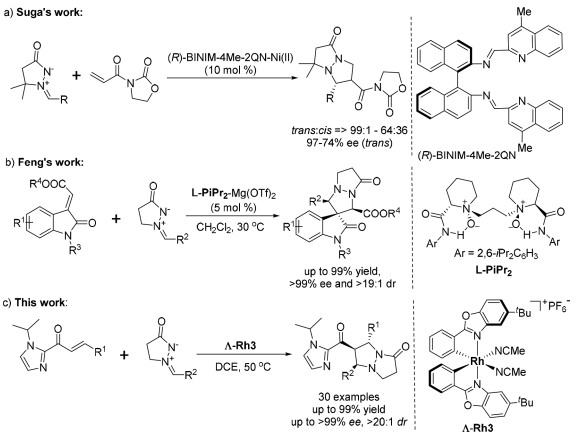


ABSTRACT: An efficient asymmetric 1,3-dipolar cycloaddition of *α,β*-unsaturated 2-acyl imidazoles with *N,N′*-cyclic azomethine imines catalyzed by a chiral-at-metal rhodium complex is reported. The corresponding *N,N′*-bicyclic pyrazolidine derivatives with three contiguous tertiary stereocenters were obtained in good yields (up to 99%) with excellent stereoselectivities (>20:1 dr and >99% ee). Remarkably, as little as 0.5 mol % of a chiral Rh(III) complex can promote a gram-scale reaction with excellent yield and enantioselectivity.

1,3-Dipolar cycloaddition (1,3-DC) has been a powerful method for construction of a variety of five-membered heterocycles from relatively simple precursors.[1] 1,3-Dipoles, such as azomethine ylides,[2] nitrones,[3] nitrile imines,[4] and nitrile oxides,[5] have been employed for the cycloaddition reactions. Since the first example of a catalytic asymmetric 1,3-DC by use of *N,N′*-cyclic azomethine imines[6,7] as suitable substrates in 2003 as reported by Fu's group, a variety of organo[8] and organometallic catalysts[9] have been developed for enantioselective construction of *N,N′*-bicyclic pyrazolidine derivatives with biological activities.[10] Aside from well-developed chiral ligand/Cu catalytic systems,[9a−k] other asymmetric organometallic catalysts have been explored.[9l−n] In 2007, Suga and coworkers reported an asymmetric 1,3-DC reaction between fused azomethine imines and 3-acryloyl-2-oxazaolidinones catalyzed by a chiral BINIM−Ni(II) complex (Scheme 1a).[9l] Recently, Feng's group developed a highly efficient chiral *N,N′*-dioxide−$Mg(OTf)_2$ complex catalyzed asymmetric 1,3-DC reaction between methyleneindolinones and *N,N′*-cyclic azomethine imines for synthesis of pyrazolidine products with contiguous quaternary−tertiary stereocenters (Scheme 1b).[9n] Despite these impressive achievements, the development of efficient asymmetric organometallic catalysts, which could solve long-standing problems such as high catalyst loading (usually 5−10 mol %) and long reaction time, is still in great demand.

Chiral-at-metal rhodium complexes developed by the Meggers group have proven to be powerful chiral Lewis acid catalysts in which the metal center serves not only as a Lewis acid but also as the exclusive source of chirality.[11] A variety of asymmetric transformations have been successfully achieved with high catalytic efficiency and enantioselectivity.[12] As a continuation of our interest in chiral-at-metal rhodium catalysis,[13] herein we report a highly efficient asymmetric 1,3-DC reaction of *α,β*-unsaturated 2-acyl imidazoles with *N,N′*-cyclic azomethine imines catalyzed by chiral-at-metal Rh(III) complexes, affording the *N,N′*-bicyclic pyrazolidine derivatives with three contiguous tertiary stereocenters (Scheme 1c).

Scheme 1. Asymmetric 1,3-Dipolar Cycloadditions Catalyzed by Metal Complexes



We started our studies on this topic by investigating the reaction of *α,β*-unsaturated 2-acyl imidazole **1a** with *N,N′*-cyclic azomethine imine **2a** in the presence of chiral-at-metal Rh(III) complex **Λ-Rh1**[13g] (2 mol %), which was synthesized by our

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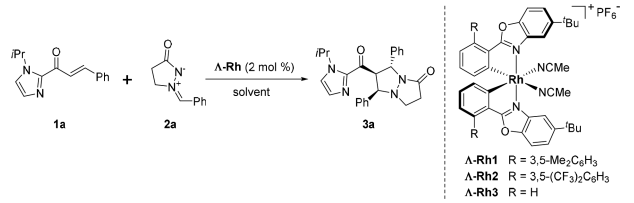
group. To our delight, the reaction proceeded smoothly in 1,2-dichlorethane (DCE) to afford the desired product **3a** in 99% yield with >20:1 dr and 82% ee (Table 1, entry 1). Encouraged

Table 1. Optimization of the Reaction Conditions[a]



entry	catalyst	solvent	time (h)	yield[b] (%)	dr[c]	ee[d] (%)
1	**Λ-Rh1**	DCE	16	99	>20:1	82
2	**Λ-Rh2**	DCE	16	99	>20:1	90
3	**Λ-Rh3**	DCE	16	99	>20:1	99
4	**Λ-Rh3**	THF	16	99	>20:1	99
5	**Λ-Rh3**	toluene	16	99	>20:1	99
6	**Λ-Rh3**	MeCN	36	98	>20:1	97
7[e]	**Λ-Rh3**	CH_2Cl_2	36	98	>20:1	99
8	none	DCE	24	0	nd	nd

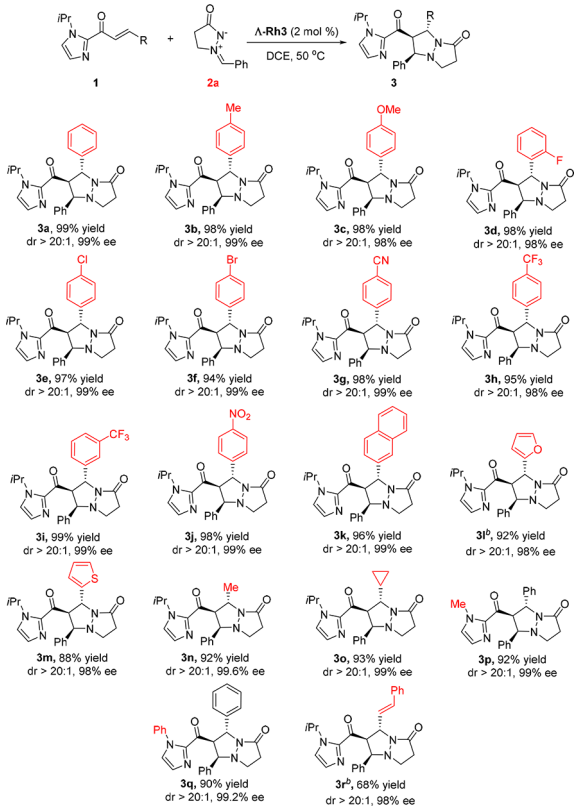
[a]Reaction conditions: **1a** (0.20 mmol), **2a** (0.24 mmol), catalyst (2.0 mol %) in solvent (0.4 mL) at 50 °C under argon atmosphere. [b]Isolated yield. [c]Determined by crude ^{1}H NMR. [d]Chiral HPLC analysis. [e]At 30 °C. nd = not determined.

by this promising result, we examined other chiral Rh(III) complexes. When **Λ-Rh2**[13f] was used, the enantioselectivity was improved to 90% (entry 2). Gratifyingly, **Λ-Rh3** developed by Meggers' group[12a] was the best one in terms of enantioselectivity, giving the desired product in 99% yield with 99% ee and >20:1 dr (entry 3). Various solvents such as THF, toluene, MeCN, and CH_2Cl_2 also could provide comparable results (entries 4−7). A control experiment in the absence of a catalyst failed to provide any product, thereby demonstrating that this reaction crucially depends on the chiral-at-metal Rh(III) complexes (entry 8).

With the optimized reaction conditions in hand (Table 1, entry 3), we next investigated the scope of *α,β*-unsaturated 2-acyl imidazoles in this reaction (Scheme 2). The introduction of electron-donating and electron-withdrawing groups on the phenyl ring of *α,β*-unsaturated 2-acyl imidazoles had little influence on both the yields and stereoselectivities. The desired products **3b−j** were obtained in high yields (94−99%) with excellent stereoselectivities (98−99% ee and >20:1 dr). Naphthyl-based **1k** and heteroaromatic **1l,m** were all well converted in good yields with excellent stereoselectivities (98−99% ee and >20:1 dr, **3k−m**). Methyl- or cyclopropyl-substituted *α,β*-unsaturated 2-acyl imidazoles were also tolerated well, affording products **3n** and **3o** in good yields with excellent ee (>20:1 dr, 99−99.6% ee). Moreover, replacing the *N*-isopropylimidazole with *N*-methylimidazole or *N*-phenylimidazole had no significant influence on the reactivity and selectivity (**3p−q**). Styryl-substituted *α,β*-unsaturated 2-acyl imidazole **1r** was also suitable substrate for this reaction, although moderate yield was obtained (68% yield, 98% ee, **3r**).

Further investigation of the substrate scope of *N,N′*-cyclic azomethine imines was carried out. As illustrated in Scheme 3, azomethine imine with an electron-donating methyl or methoxy group on the phenyl ring could react smoothly with **1a** to afford the corresponding products (**4a,b**). Azomethine imines equipped with halogen atoms were all converted in good

Scheme 2. Substrate Scope of *α,β*-Unsaturated 2-Acyl Imidazoles.[a]



[a]Reaction conditions: **1a−r** (0.20 mmol), **2a** (0.24 mmol), and **Λ-Rh3** (2.0 mol %) in DCE (0.4 mL) at 50 °C under argon atmosphere. All isolated yields were based on substrate **1**. The ee values were determined by HPLC analysis using chiral stationary phase. The dr values were detected by crude ^{1}H NMR. [b]3.0 mol % of **Λ-Rh3** was used.

yields with excellent ee (96−99% yields, 98−99.4% ee, **4c−f**). Azomethine imines bearing a strong electron-withdrawing substituent (−CF_3 or −NO_2) on the phenyl ring could react with **1a** with excellent stereoselectivities, albeit with a slightly lower yield (87−88% yield, **4g,h**). Heteroaromatic and 1-naphthyl-substituted azomethine imines were tolerable under the optimal reaction conditions, delivering the desired products **4i−k** in 76−99% yields with satisfactory stereoselectivities (94−98% ee, 10:1 to >20:1 dr). To our delight, replacing the aromatic group with an aliphatic cyclohexyl group on an azomethine imine could also provide the corresponding product **4l** in 74% yield with 93% ee and >20:1 dr, although 4 mol % of **Λ-Rh3** was needed.

To demonstrate the synthetic utility of the current protocol, a gram-scale reaction of **1a** (1.06 g, 4.4 mmol) and azomethine imine **2a** (919.8 mg, 5.28 mmol) was conducted in the presence of 1.0 mol % **Λ-Rh3** (Scheme 4a). Gratifyingly, the reaction proceeded smoothly to afford **3a** in 98% yield (1.79 g), with 99% ee and >20:1 dr. Remarkably, when as low as 0.5 mol % of **Λ-Rh3** was employed, the product **3a** still could be obtained in 98% yield with excellent enantioselectivity (96% ee). The gram-scale reaction exhibits extraordinary reactivity and stereoselectivity of a chiral Rh(III) complex on the title reactions. Moreover, the product **3a** could be converted into optically active alcohol **5** and aldehyde **6** by reduction and

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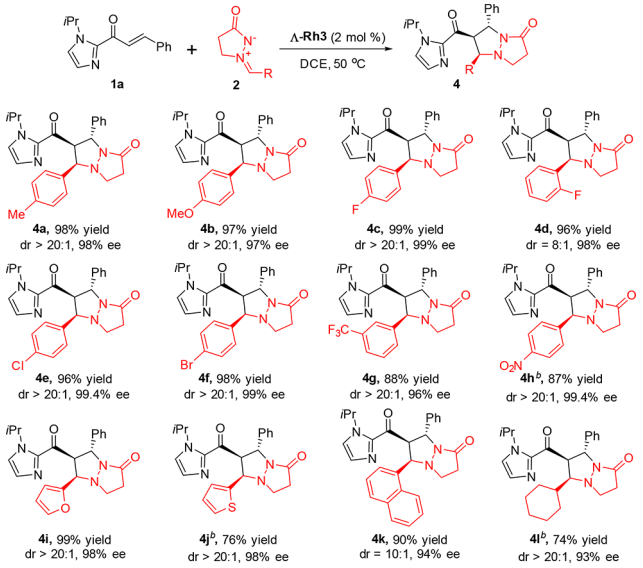
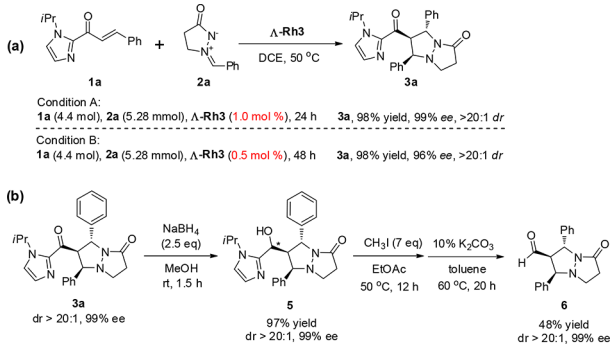
Scheme 3. Substrate Scope of *N,N′*-Cyclic Azomethine imines[a]



[a]Reaction conditions: **1a** (0.20 mmol), **2b−m** (0.24 mmol) and **Λ-Rh3** (2.0 mol %) in DCE (0.4 mL) at 50 °C under argon atmosphere. All isolated yields were based on substrate **1**. The ee values were determined by HPLC analysis using chiral stationary phase. The dr values were detected by crude ^{1}H NMR. [b]4.0 mol % of **Λ-Rh3** was used.

Scheme 4. Gram-Scale Reaction and Synthetic Transformations



further removal of imidazole moiety without any loss in enantiomeric excess (Scheme 4b).

On the basis of the previous investigations,[12a] a model of stereoselective control is proposed (Figure 1). The *α,β*-unsaturated 2-acyl imidazole can be activated by the chiral Rh(III) complex through bidentate N,O-coordination. The *Si*-face of the coordinated substrate is effectively shielded by one of the *tert*-butyl groups. The highly selective approach of the *N,N′*-cyclic azomethine imine from the *Re*-face of the coordinated substrate leads to the desired product. The absolute configuration of **3e** was determined to be (1*S*,2*R*,3*S*) by X-ray crystallographic analysis, and other products were assigned by analogy (Figure 1, for details, see the Supporting Information).

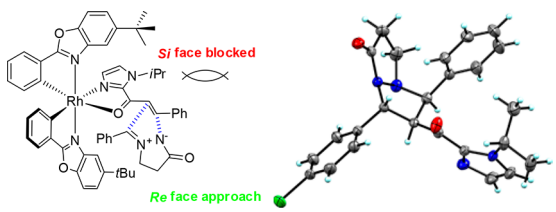


Figure 1. Proposed model and the X-ray structure of **3e** with thermal ellipsoids shown at the 35% probability level.

In conclusion, we have developed a highly efficient asymmetric 1,3-dipolar cycloaddition of *α,β*-unsaturated 2-acylimidazoles with *N,N′*-cyclic azomethine imines catalyzed by chiral-at-metal rhodium complexes, affording the *N,N′*-bicyclic pyrazolidine derivatives with three contiguous tertiary stereocenters in up to 99% yields, >20:1 dr, and >99% ee. The reaction features simple operation, low catalyst loading, wide substrate scope, and mild reaction conditions. Remarkably, this protocol exhibits extraordinary advantages in terms of reactivity and enantioselectivity, given the fact that as little as 0.5 mol % of **Λ-Rh3** can realize the title reaction on gram scale, yielding the desired product with excellent stereoselectivity.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b01264.

Experimental procedures, characterization data, and ^{1}H and ^{13}C NMR spectra and HPLC chromatograms for obtained compounds (PDF)

Accession Codes

CCDC 1835990 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.

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Notes

The authors declare no competing financial interest.

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