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Highly Enantioselective Ruthenium-Catalyzed Cascade Double Reduction Strategy: Construction of Structurally Diverse Julolidines and Their Analogues

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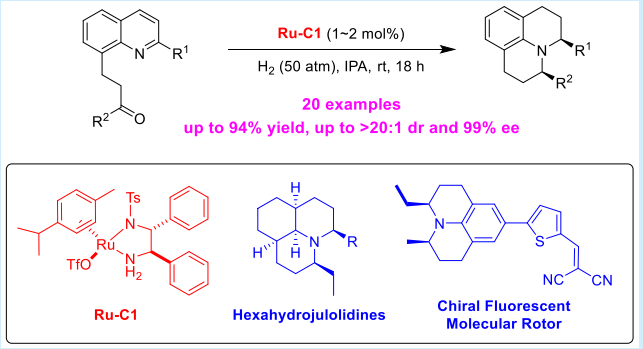
ABSTRACT: A direct and facile construction of optically pure julolidine derivatives through ruthenium-catalyzed enantioselective cascade hydrogenation and reductive amination of 2-(quinolin-8-yl)ethyl ketones has been developed. By means of this protocol, various chiral julolidine compounds were obtained in high isolated yields (up to 94%) with excellent diastereoselectivities (up to >20:1 dr) and enantioselectivities (up to 99% ee) under mild conditions. Furthermore, the synthetic practicality of this protocol was illustrated by the preparation of hexahydrojulolidines and a chiral fluorescent molecular rotor.



Julolidine and its derivatives, as one of the important classes of heteropolycyclic compounds, have attracted continued attention from the scientific community because of their many applications in the photochemical industry as fluorescent dyes and also in biological systems (Figure 1).[1] Over the past several decades, numerous strategies and methods have been developed for the synthesis of julolidine derivatives.[2,3] However, the catalytic asymmetric synthesis of optically active julolidine derivatives is less studied.[3] In 2010, Gong and co-workers realized the construction of chiral julolidine derivatives through an enantioselective three-component cascade Povarov cycloaddition and intramolecular hydroamination reaction followed by reduction with sodium triacetoxyborohydride (Scheme 1a).[3a] Most recently, Rueping and co-workers reported a highly enantioselective synthesis of a julolidine analogue through a Brønsted acid-catalyzed aza-Diels−Alder reaction between in situ-generated aza-*o*-quinone methide and styrene (Scheme 1b).[3b] These approaches, however, still suffer from low diastereoselectivity and/or the lack of substrate generality. Therefore, the development of step-efficient asymmetric approaches for the rapid and direct generation of structurally diverse chiral julolidine derivatives is highly desirable and still a big challenge.

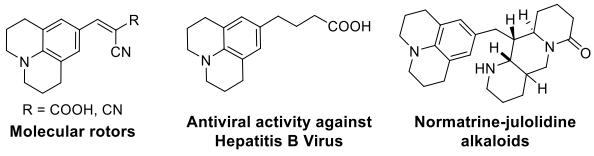


Figure 1. Some examples and applications of julolidine derivatives.

Recently, we found that chiral cationic Ru(diamine) complexes[4] are very efficient catalysts for the enantioselective hydrogenation of N-containing heteroarenes and ketimines with high activities and excellent enantioselectivities.[5,6] More recently, we demonstrated that this catalytic system can efficiently catalyze the enantioselective cascade hydrogenation/reductive amination of quinolinyl ketones and quinoxalinyl ketones, affording various chiral quinolizidine and indolizidine derivatives in high yields and optical purity.[7] Inspired by these results, we proposed that 2-(quinolin-8-yl)ethyl ketones **1** could be directly converted into optically pure julolidine derivatives **2** through a cascade double reduction strategy (Scheme 1c). Herein we describe a protocol involving ruthenium-catalyzed highly enantioselective cascade hydrogenation and reductive amination of 2-(quinolin-8-yl)ethyl ketones, providing facile access to structurally diverse chiral julolidine derivatives.

In our initial studies, the cascade reaction of **1a** catalyzed by 1.0 mol % (*R*,*R*)-**C1** was selected as the model reaction for optimization of the reaction conditions (Table 1 and Tables S1 and S2 in the Supporting Information). To our delight, the

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Scheme 1. Strategies for Enantioselective Synthesis of Chiral Julolidines

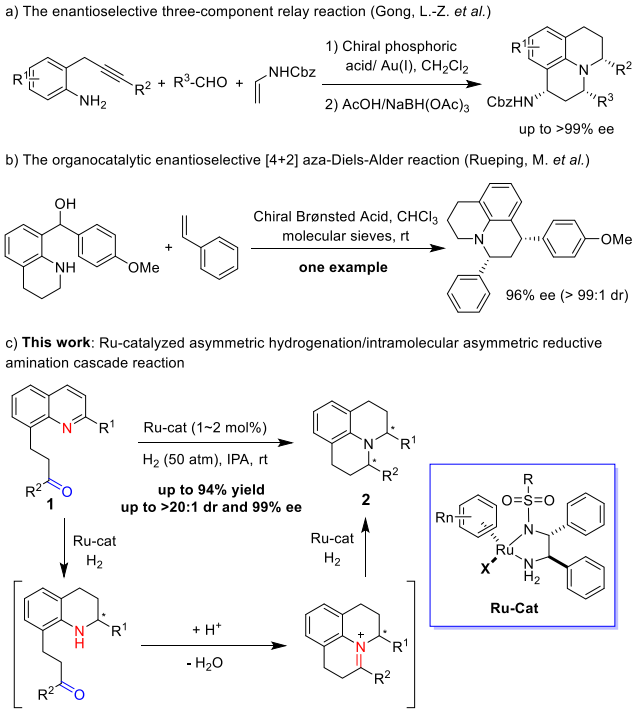
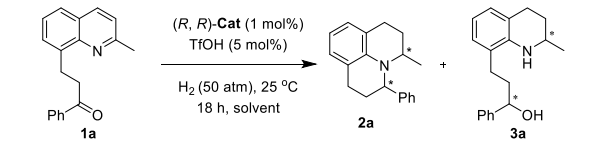
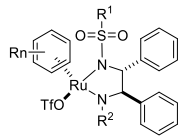


Table 1. Optimization of the Reaction Conditions[a]





(*R*,*R*)-**C1**-**C6**

(*R*,*R*)-**C1**: R^1 = 4-$CH_3C_6H_4$; R^2 = H; η^6-arene = *p*-cymene
(*R*,*R*)-**C2**: R^1 = CH_3; R^2 = H; η^6-arene = *p*-cymene
(*R*,*R*)-**C3**: R^1 = CH_3; R^2 = H; η^6-arene = mesitylene
(*R*,*R*)-**C4**: R^1 = CH_3; R^2 = H; η^6-arene = hexamethylbenzene
(*R*,*R*)-**C5**: R^1 = CH_3; R^2 = H; η^6-arene = benzene
(*R*,*R*)-**C6**: R^1 = 4-$CH_3C_6H_4$; R^2 = CH_3; η^6-arene = *p*-cymene

entry	solvent	catalyst	conv. (%)[b]	2a:3a[b]	dr[b]	ee of 2a (%)[c]
1	MeOH	(*R*,*R*)-**C1**	>95	78:22	9:1	99
2	EtOH	(*R*,*R*)-**C1**	>95	87:13	9:1	98
3	IPA	(*R*,*R*)-**C1**	>95	95:5	9:1	98
4	THF	(*R*,*R*)-**C1**	<5	–	–	–
5	IPA	(*R*,*R*)-**C2**	88	94:6	6:1	95
6	IPA	(*R*,*R*)-**C3**	77	60:40	6:1	96
7	IPA	(*R*,*R*)-**C4**	88	93:7	9:1	97
8	IPA	(*R*,*R*)-**C5**	38	83:17	10:1	90
9	IPA	(*R*,*R*)-**C6**	>95	88:12	9:1	99
10[d]	IPA	(*R*,*R*)-**C1**	>95	88:12	12:1	99
11[e]	IPA	(*R*,*R*)-**C1**	65	90:10	5:1	98
12[f]	IPA	(*R*,*R*)-**C1**	>95	94:6	12:1	99

[a]Reactions were carried out on a 0.1 mmol scale using (*R*,*R*)-Ru catalyst (1.0 mol %) and TfOH (5 mol %) in the solvent (0.5 mL) under an atmosphere of H_2 (50 atm) at 25 °C for 18 h. [b]Determined by ^{1}H NMR analysis of the crude products. [c]Determined by HPLC analysis using a chiral column. [d]In the absence of TfOH. [e](*R*,*R*)-**C1** (0.2 mol %). [f](*R*,*R*)-**C1** (2.0 mol %).

cascade reaction in methanol proceeded smoothly, affording the desired chiral julolidine **2a** in full conversion with good diastereoselectivity and excellent enantioselectivity (entry 1). High reactivity could only be observed in alcoholic solvents (Table 1, entries 1−4, and Table S1), and isopropanol (IPA) was found to be the optimal solvent in terms of both chemoselectivity and stereoselectivity (entry 3). Moreover, various Ru(diamine) catalysts were screened (entries 5−9), and (*R*,*R*)-**C1** proved to be the optimal catalyst (entry 3). Notably, slightly decreased chemoselectivity was observed under TfOH-free conditions, indicating that the acid additive enhanced the reductive amination process (entry 10). Further studies were made to adjust the hydrogen pressure and reaction temperature. The best result was achieved under 50 atm H_2 at 25 °C (Table S2). When the catalyst loading was reduced to 0.2 mol %, identical enantioselectivity but decreased conversion and diastereoselectivity were observed (entry 11). Increasing the catalyst loading to 2 mol % slightly improved the enantioselectivity (99% ee) and diastereoselectivity (12:1 dr) (entry 12). Therefore, the optimal conditions were identified to be the following: (*R*,*R*)-**C1** (2 mol %) as the catalyst and IPA as the solvent under 50 atm H_2 at 25 °C for 18 h.

With the optimal conditions in hand, various 2-(2-alkylquinolin-8-yl)ethyl aryl ketones were investigated. In general, all of the cascade reactions proceeded smoothly, affording the desired products **2a**−**m** in high isolated yields with good to excellent diastereoselectivities (7:1 to >20:1 dr) and excellent enantioselectivities (97−99% ee). It is noteworthy that electron-donating and electron-withdrawing substituents at the *para* position of the phenyl ring exhibited little effect on either the reactivity or the selectivity (**2a**−**e**). Moreover, a substrate bearing a methoxy group at the 2-position of the phenyl ring gave similar excellent enantioselectivity but slightly lower diastereoselectivity (**2g** vs **2c** and **2f**). Interestingly, improved diastereoselectivities (10:1 to >20:1 dr) were observed with alkyl chains (R_1) of increasing length from a methyl group to a 2-phenylethyl group (**2a** versus **2i**−**k**). In addition, the absolute configuration of chiral julolidines **2a**−**m** was determined to be (3*R*,5*R*) by single-crystal X-ray analysis of product **2d** (Figure S1).

Subsequently, the tandem reaction of 2-(2-alkylquinolin-8-yl)ethyl alkyl ketones was also examined (Scheme 2, **2n**−**s**). To our delight, the substrates bearing linear alkyl groups also gave excellent results. Various chiral dialkyl-substituted julolidine products were obtained in high isolated yields (80%−92%) with excellent levels of diastereoselectivity (17:1 to >20:1 dr) and enantioselectivity (98−99% ee) (**2n**−**r**). However, a slightly lower enantioselectivity (93% ee) was observed when a sterically hindered cyclopropyl group was introduced as R_2 (**2s**). Encouraged by these results, we also studied the challenging substrate 2-(2-phenylquinolin-8-yl)-ethyl tolyl ketone (**1t**). Unfortunately, only moderate conversion (57%) and low enantioselectivity (13% ee) were observed under the same conditions (Table S3, entry 1). Upon further optimization of the reaction parameters (Tables S3 and S4), the reduced product, diaryl-substituted julolidine **2t**, was obtained in 85% isolated yield with >20:1 dr and 61% ee using 10 mol % (*R*,*R*)-**C5** in the presence of 50 mol % TfOH.

To further exemplify the synthetic applicability of the above-developed cascade strategy, hexahydrojulolidine derivatives and a chiral fluorescent molecular rotor were prepared (Schemes 3 and 4). Under the above optimal reaction conditions, the cascade reactions of **1a** and **1n** on scales of 605 mg and 632 mg, respectively, proceeded smoothly,

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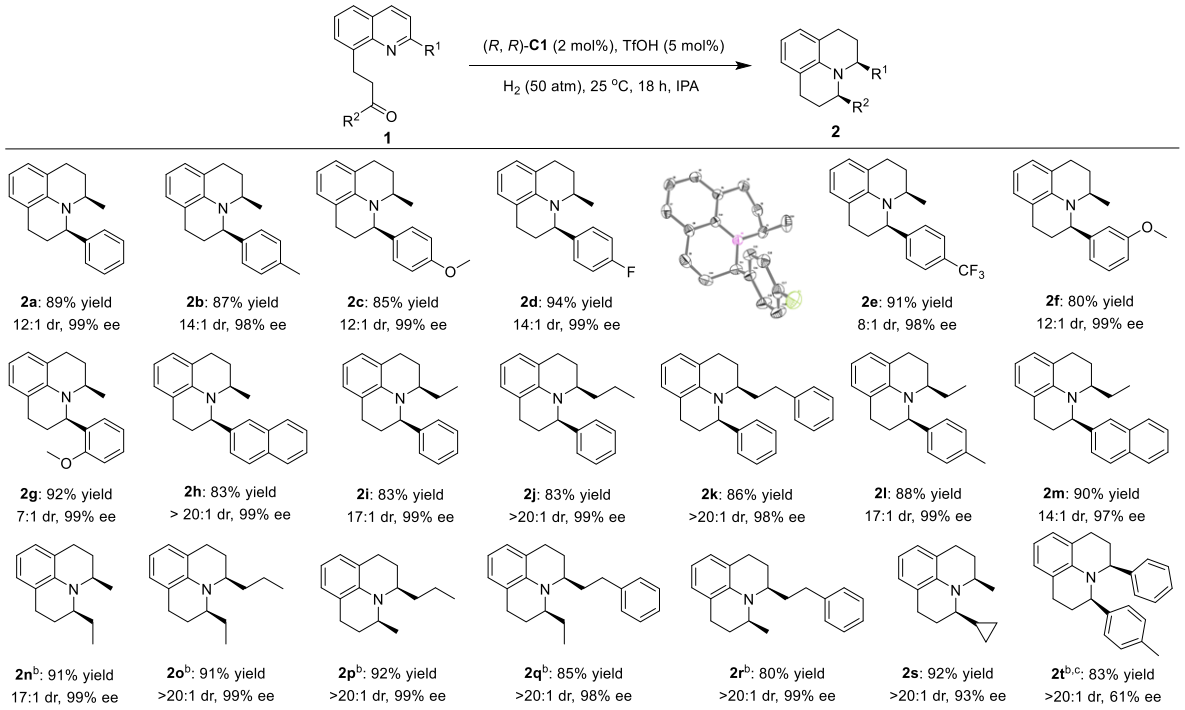
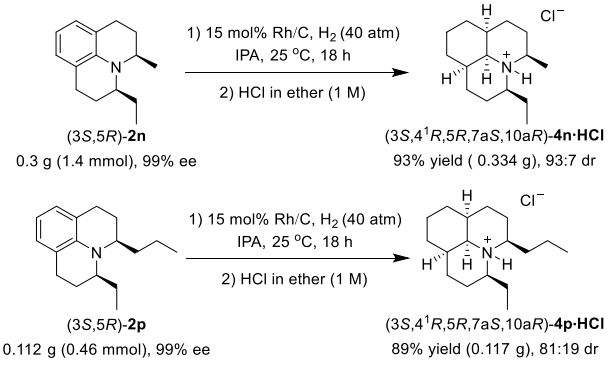
Scheme 2. Synthesis of Chiral Julolidines: Substrate Scope[a]

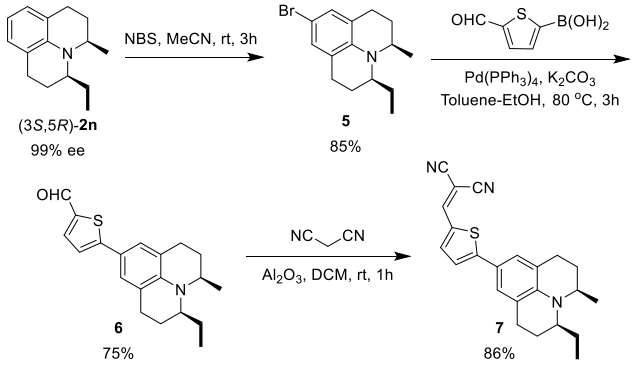


[a]Reaction conditions: substrate (0.1 mmol) in IPA (0.6 mL), (*R*,*R*)-**C1** (2.0 mol %), H_2 (50 atm), stirred at 25 °C for 18 h. Isolated yields of both reduced isomers are shown. The ee values were determined by HPLC with a chiral stationary phase. [b]The isolated yield of the main isomer is shown. [c](*R*,*R*)-**C5** (10.0 mol %), TfOH (50 mol %).

Scheme 3. Synthesis of Chiral Hexahydrojulolidine Derivatives



Scheme 4. Synthesis of Chiral Molecular Rotor 7



affording the corresponding chiral julolidines **2a** and **2n** with similar excellent results (Scheme S2).

Hexahydrojulolidine is the basic ring skeleton in various biologically active *Lycopodium* alkaloids.[8] However, efficient synthetic methods for this class of polycyclic aliphatic N-heterocycles have rarely been reported to date.[9] As shown in Scheme 3, optically pure (3*S*,5*R*)-**2n** was smoothly hydrogenated in the presence of 15 mol % Rh/C in IPA,[10] followed by treatment with HCl to afford the chiral hexahydrojulolidine **4n·HCl** in 93% isolated yield with high diastereoselectivity (93:7 dr). Similarly, chiral **4p·HCl** was also obtained in 89% yield with good diastereoselectivity (81:19 dr). Furthermore, the absolute configuration of chiral product **4n** was determined to be (3*S*,4¹*R*,5*R*,7a*S*,10a*R*) by single-crystal X-ray analysis of the hydrochloride of **4n** (Figure S2).

Fluorescent molecular rotors have been attracting much attention because of their ability to serve as viscosity sensors in various media.[11] To our delight, by means of this cascade strategy a julolidine-based chiral fluorescent molecular rotor could be easily synthesized via a four-step procedure starting from a readily available quinoline derivative. As illustrated in Scheme 4, bromination of chiral julolidine (3*S*,5*R*)-**2n** by NBS in MeCN gave bromo compound **5** in 85% isolated yield. Then the Pd(0)-catalyzed Suzuki cross-coupling reaction of **5** with 5-formyl-2-thiopheneboronic acid followed by Knoevenagel condensation with malononitrile afforded the target chiral fluorescent molecular rotor **7** in 65% yield over the two steps. We believe that this new chiral fluorescent molecular rotor **7** has the potential to serve as a viscosity sensor in chiral active materials.

In summary, a step-efficient protocol for the construction of structurally diverse and enantioenriched julolidine derivatives from 2-(quinolin-8-yl)ethyl ketones through Ru-catalyzed cascade enantioselective hydrogenation and reductive amination has been developed. Various chiral julolidine derivatives

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were obtained in high isolated yields with excellent diastereoselectivities and enantioselectivities. Furthermore, the utility of this protocol was showcased by the preparation of hexahydrojulolidines and a new chiral fluorescent molecular rotor.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c00444.

Optimization tables, experimental procedure, details of synthesis and characterization data for all of the substrates and products, and crystallographic information for **2d** and **4n·HCl** (PDF)

Accession Codes

CCDC 1974650 and 1974652 contain 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, U.K.; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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