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Ruthenium-Catalyzed Enantioselective Hydrogenation of Hydrazones

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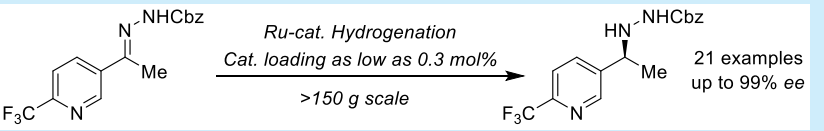
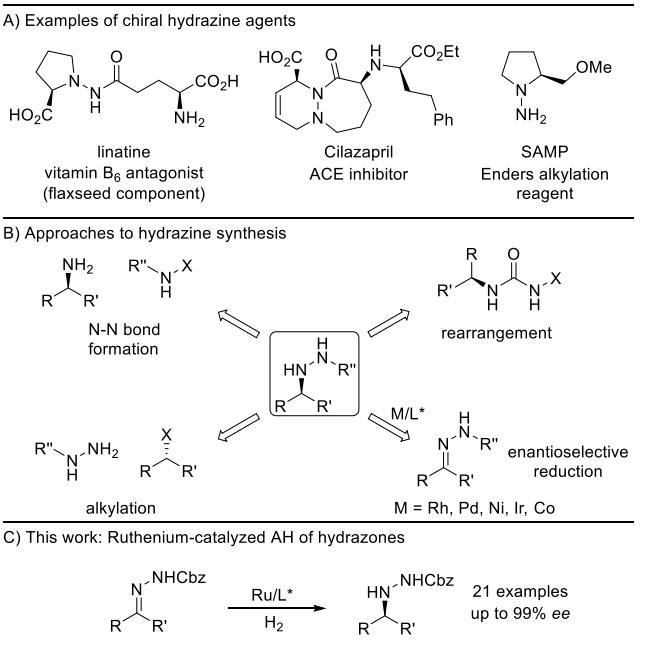


ABSTRACT: Prochiral hydrazones undergo efficient and highly selective hydrogenation in the presence of a chiral diphosphine ruthenium catalyst, yielding enantioenriched hydrazine products (up to 99% *ee*). The mild reaction conditions and broad functional group tolerance of this method allow access to versatile chiral hydrazine building blocks containing aryl bromide, heteroaryl, alkyl, cycloalkyl, and ester substituents. This method was also demonstrated on >150 g scale, providing a valuable hydrazine intermediate en route to an active pharmaceutical ingredient.

Since the pioneering work of Emil Fischer in the 1870s, hydrazine derivatives have become an indispensable class of molecules of great importance to modern life.[1] Such N−N bond containing compounds can be found in a wide range of applications from dyes, corrosion inhibitors and fuels, to catalysts, synthetic reagents, pharmaceuticals, and agro chemicals (Scheme 1A).[2] Nature itself has also incorporated N−N motifs in a variety of natural products resulting in unique molecular architectures and interesting biological activities.[3] Many of these structures contain N−N linkages attached at chiral centers, inspiring the development of new methods for their selective synthesis.[4] Several of these approaches are highlighted in Scheme 1B and include both C−N bond forming and N−N bond forming transformations.

Scheme 1. Synthetic Approaches towards Chiral Hydrazines



One of the most successful methods that has emerged for the synthesis of chiral hydrazines is the selective reduction of prochiral hydrazones. Early examples of this approach relied on preexisting stereocenters in order to deliver reducing agents in a diastereoselective manner.[5] A breakthrough in the field was achieved by Burk, who in 1992 reported the first example of enantioselective reduction of hydrazones, utilizing a rhodium/DuPhos catalyst.[6] Since this initial report, many excellent examples of enantioselective hydrazone reduction have been disclosed (Scheme 1B). Following the work of Burk, many of these reports employ the use of rhodium[7] based catalysts, with fewer examples of palladium,[8] nickel,[9] iridium,[10] and cobalt[11] based catalysts. Despite the great success of ruthenium catalysts in related imine reduction chemistry,[12] to the best of our knowledge there are no examples of utilizing group 8 metals in hydrazone reduction. Herein, we report the first example of enantioselective ruthenium-catalyzed hydrogenation of hydrazones.

As part of a recent campaign toward a pharmaceutical intermediate, an asymmetric preparation of large amounts of

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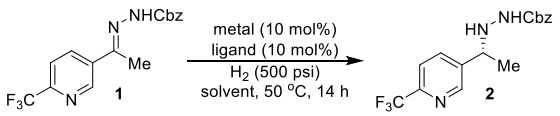
hydrazine **2** was required (Table1). Due to the relative ease of deprotection, initial focus was placed on targeting an

Table 1. Selected Screening Data for the Enantioselective Reduction of 1[a]



entry	solvent	metal[b]	ligand	conv (%)[c]	*ee* (%)[c]
1	MeOH	Rh	T002-1	7	22
2	MeOH	Rh	J011-1	96	74
3	MeOH	Rh	J004-1	94	86
4[d]	MeOH	Rh	(*R*)-MP2-SegPhos	93	88
5	MeOH	Rh	(*S*,*S*)-iPr-DiPAMP	91	81
6	MeOH	Ru	W003-1	>98	92
7	MeOH	Ru	W017-1	>98	97
8	MeOH	Ru	W022-1	>98	97
9	MIBK	Ru	W022-1	>98	99
10[e,f]	MIBK	Ru	W022-1	>98	98
11[e,g]	MIBK	Ru	W022-1	>98	97

[a]Conditions: metal precursor, ligand, solvent (0.02M), 500 psi H_2, 50 °C. [b]Rh = $(NBD)_2RhBF_4$, Ru = $(methallyl)_2Ru(cod)$ + $HBF_4{-}OEt_2$ (2 equiv to Ru). [c]As determined by chiral SFC analysis at 210 nm. Absolute configuration is (*R*) unless otherwise noted. [d](*S*) product obtained. [e]0.2 M in MIBK (methylisobutylketone). [f]1.0 mol % Ru/ligand. [g]0.3 mol % Ru/ligand.

enantioselective reduction of Cbz protected hydrazone **1**, which was prepared from the commercially available ketone and subjected to screening conditions (Table 1). Initial attempts were inspired by the work of Tan and Yoshikawa[7e] and centered around the use of rhodium/chiral phosphine based catalysts (entries 1−5). Unfortunately, these conditions failed to yield sufficiently high levels of enantioselectivity, with the best result giving 88% *ee* when employing (*R*)-MP2-SegPhos (Figure 1) in combination with $(NBD)_2RhBF_4$ at 50

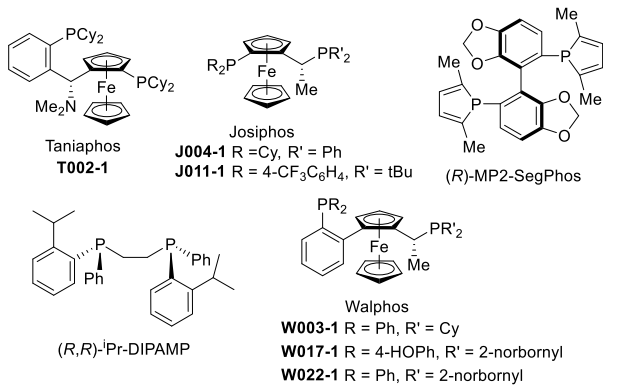
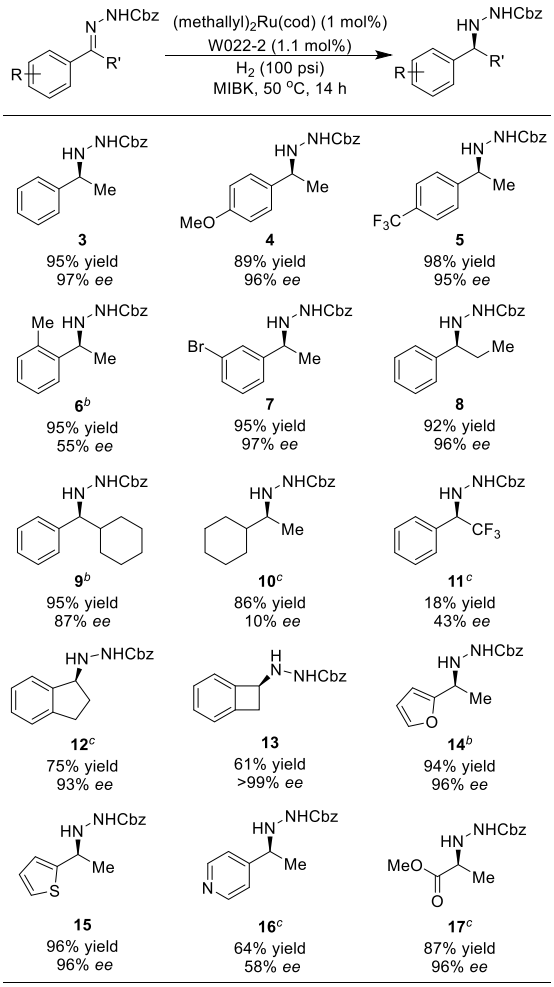


Figure 1. Chiral diphosphine ligands used in Table 1.

°C in methanol (entry 4). It was not until screening ruthenium/chiral phosphine based catalysts that high levels of enantioselectivity were obtained, with Walphos[13] and $(methallyl)_2Ru(cod)$[14] combinations performing most effectively (entries 6−8). Further optimization of reaction conditions identified MIBK[15] as the ideal solvent together with Walphos derivative W022 as ligand, affording full conversion to desired product **2** with 99% *ee* (entry 9).[16] Importantly, the catalyst loading could be lowered to 0.3 mol % and still maintain efficient hydrazone reduction (entry 11).

With a highly active and enantioselective catalyst system in hand, the scope of ruthenium-catalyzed hydrazone reduction was examined (Table 2). Notably, both electron-rich and

Table 2. Scope of Enantioselective Hydrogenation of Hydrazones[a]



[a]Conditions: $(methallyl)_2Ru(cod)$ (1 mol %), W022-2 (1.1 mol %), $HBF_4{-}OEt_2$ (2 mol %), MIBK (0.2 M), 50 °C, 100 psi H_2 unless otherwise noted, yield refers to assay yield of reaction mixture determined by quantitative HPLC analysis. [b]200 psi H_2. [c]500 psi H_2.

electron-poor aryl substituents are well tolerated, fully converting to the desired hydrazides **4** and **5** with excellent levels of enantioselectivity. This is in stark contrast to rhodium-catalyzed hydrazone hydrogenations, which were shown by Burk to exhibit a strong electronic dependence on the aryl substituent with deficient groups consistently outperforming donating groups.[6b] Sterically challenging *ortho*-methyl containing substrate **6** and aryl bromide **7** were reduced in high yield. Only moderate enantioselectivity for formation of **6** was achieved, possibly due to a conformational change induced by having an ortho substituent. Crucially, **7** exhibited no signs of reductive dehalogenation. Examination of other substitution patterns revealed both ethyl substitution (**8**) and cyclohexyl substitution (**9**)[17] gave efficient reaction, highlighting the ability of the catalyst to distinguish between aryl groups and

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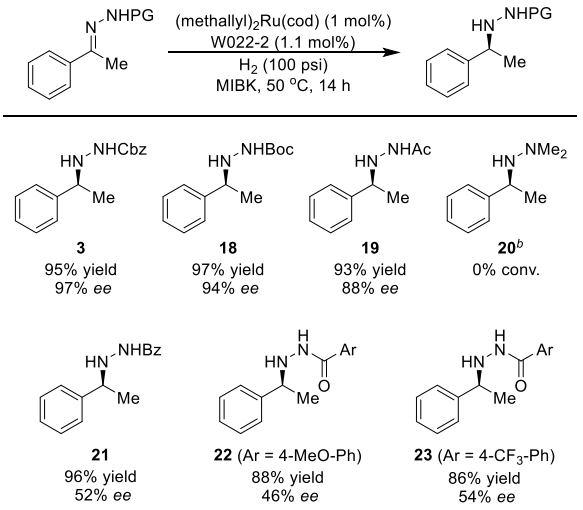
groups larger than methyl. Interestingly, the selectivity of the reaction decreases significantly when both hydrazone substituents are alkyl with hydrazine **10** being produced with only 10% *ee*. Previously, work by Zhou has shown trifluoromethyl-substituted hydrazones to undergo selective reduction in the presence of chiral Pd-catalysts under forcing condtions.[8b8d] Notably, the formation of trifluoromethyl-substituted hydrazine **11** proved difficult for the ruthenium catalyst, achieving only an 18% conversion and 43% *ee*.

Additionally, cyclic hydrazones were examined, giving hydrazine products **12** and **13** in moderate to good yields and with excellent enantioselectivity. Substrates containing heteroaryl groups also underwent smooth reduction, affording enantioenriched hydrazines containing furan (**14**), thiophene (**15**), and pyridine (**16**). Significantly, the ruthenium catalyst is tolerant of thiophene which can often poison transition metal catalysts. The presence of a basic pyridine moiety appears to adversely effect catalyst efficiency with only moderate yield and selectivity obtained for **16**. It was reasoned that the unhindered pyridine could participate as an unwanted ligand for the ruthenium catalyst and thereby inhibit catalysis. Attempts to quench this interference by treating the substrate with a stoichiometric equivalent of HBF_4 proved unsuccessful, leading to no conversion to **16**. α-Hydrazino acids are interesting analogs of amino acids which have garnered significant attention as synthetic targets.[18] Reduction of an α-ester hydrazone proved successful, and α-hydrazino-ester **17** was prepared in good yield and with excellent enantioselectivity.

In order to examine the scope and effect of the hydrazone protecting groups in the hydrogenation reaction, analogs of acetophenone-derived hydrazone were prepared and subjected to the standard conditions (Table 3). Efficient reduction was achieved when carbamate protecting groups were employed with only a slight decrease in enantioselectivity going from Cbz (**3**) to Boc (**18**) protected hydrazines. Acetyl protected hydrazone was also reduced effectively by the ruthenium catalyst system, generating hydrazine **19** in high yield but with slightly diminished enantioselectivity. Of note, simple alkyl-substituted hydrazones do not undergo reduction, with no conversion to the dimethyl substituted target **20**, possibly due to unproductive catalyst binding by this basic substrate.

Table 3. Examination of Additional Protecting Groups[a]

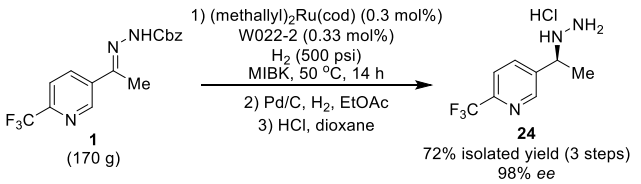


[a]Conditions: $(methallyl)_2Ru(cod)$ (1 mol %), W022−2 (1.1 mol %), $HBF_4{-}OEt_2$ (2 mol %), MIBK (0.2 M), 50 °C, 100 psi H_2 unless otherwise noted, yield refers to assay yield of reaction mixture determined by quantitative HPLC analysis. [b]500 psi H_2 and 5 mol % catalyst.

The vast majority of enantioselective hydrazone reductions utilize benzoyl-protected hydrazones as substrates.[6−11] Interestingly, this commonly employed protecting group does not achieve high levels of enantioselectivity under the standard ruthenium-catalyzed conditions, leading to benzoyl-protected hydrazine **21** in good yield but with only moderate enantioselectivity. To further probe this observation, electronically differentiated benzoyl-protected hydrazones were prepared and subjected to the standard reaction conditions. Notably, both a more electron-rich benzoyl-group (**22**) and a more electron-poor benzoyl group (**23**) led to similar levels of enantioselectivity. This is in contrast to rhodium-catalyzed hydrazone hydrogenations where a strong dependence on protecting group electronics leads to large changes in enantioselectivity. Studies by Burk showed drastic changes in enantioselectivity with more donating protecting groups leading to increased selectivity, presumably due to more tightly bound chelates in the enantiodetermining step.[6b] The ruthenium-catalyzed conditions reported here appear to be largely complementary to the majority of other metal catalyzed hydrazone reductions, with the use of carbonate-based protecting groups giving the best enantioselectivities.

Access to large quantities of hydrazine **2** were required, and therefore the hydrogenation of hydrazone **1** was examined in scale-up studies (Scheme 2). Importantly, the hydrogenation proceeded smoothly with ruthenium loadings as low as 0.3 mol %, achieving full conversion with excellent enantioselectivity. Subsequent deprotection with Pd/C and conversion to the crystalline HCl salt allowed isolation of **24** without any detectable racemization.

Scheme 2. Scale-up Hydrogenation of Hydrazone 1



In conclusion, the first enantioselective ruthenium-catalyzed hydrogenation of hydrazones has been demonstrated. Mild reaction conditions permitted the use of a broad range of substrates, leading to useful chiral hydrazine building blocks with high enantioselectivities. Complementary to the majority of existing methods, easily cleaved carbamate-based protecting groups provided the best reactivity and enantioselectivity. Finally, the hydrogenation proved to be readily scalable at low catalyst loadings, enabling access to large quantities of valuable enantioenriched hydrazine intermediate **24**.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c02756.

Experimental procedures and characterization of all new compounds (PDF)

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Notes

The authors declare no competing financial interest.

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(15) Partial reduction of the MIBK solvent is observed under the standard reaction conditions. For a complete list of solvents screened, see the Supporting Information.

(16) Initial screening was performed with W022-1 ((*R*,*R*) enantiomer) and the remaining scope and scale-up with W022-2

((*S*,*S*) enantiomer). For a complete set of screening and optimization data, see the Supporting Information.

(17) Hydrazone starting material used in Table 2 corresponding to **9** is a 93:7 *trans*/*cis* mixture. Utilizing pure *trans* hydrazone, **9** is produced in 94% *ee* (*S*) and pure *cis* hydrazone gives 79% *ee* (*R*).

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