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A Michael Addition−Asymmetric Transfer Hydrogenation One-Pot Enantioselective Tandem Process for Syntheses of Chiral γ-Secondary Amino Alcohols

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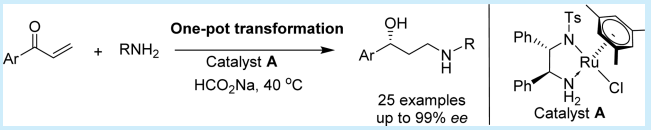
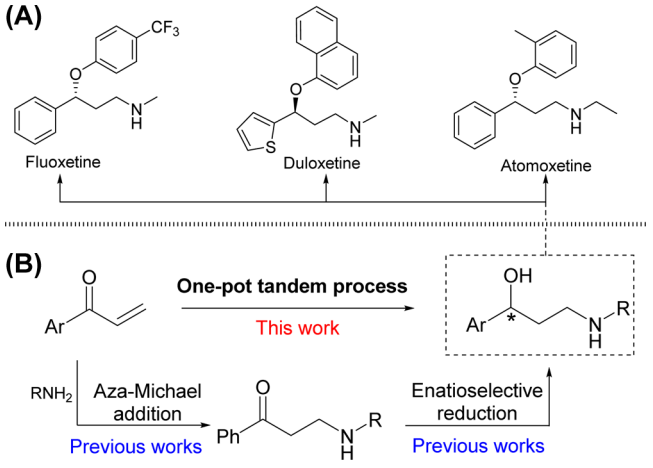
Supporting Information

ABSTRACT: An aza-Michael addition−asymmetric transfer hydrogenation tandem process for preparation of chiral γ-secondary amino alcohols has been developed. This one-pot tandem process involves an aza-Michael addition of aryl-substituted enones and amines to form aryl-substituted γ-secondary amino ketones, followed by a Ru-catalyzed asymmetric transfer hydrogenation to form aryl-substituted γ-secondary amino alcohols. An advantageous feature of this tandem reaction is that it provides various γ-secondary amino alcohols in high yields with high enantioselectivities.



One-pot tandem organic transformations, providing high-value optically pure pharmaceutical intermediates that may be used to synthesize chiral drugs, have attracted much attention.[1] A significant benefit is the atom economy of the strategy, and the minimal workup greatly reduces the amount of waste. It is especially favorable when this kind of tandem organic transformation is performed in an environmentally friendly medium. Chiral γ-secondary amino alcohols are important pharmaceutical intermediates and have been extensively applied in the preparations of various antidepressants (Scheme 1A).[2] Theoretically, a general tandem process involving aza-Michael addition of enones and amines to γ-secondary amino ketones, followed by enantioselective reduction of γ-secondary amino ketones, could be used to obtain γ-secondary amino alcohols. However, to the best of our knowledge, this direct tandem organic transformation has not hitherto been explored. Fortunately, recent explorations have led to enantioselective reductions of γ-secondary amino ketones to γ-secondary amino alcohols,[3,4] which offer potential opportunities for the development of aza-Michael addition/reduction tandem processes for one-pot organic transformations.

Scheme 1. Important Drugs Prepared by Michael−ATH Enantioselective Tandem Reaction



Surveying recent efforts in the enantioselective reductions of γ-secondary amino ketones, prominent contributions have been centered on the strategy of asymmetric hydrogenation of prochiral γ-amino ketones.[4] In earlier reports by the groups of Achiwa and Zhang, the chiral Rh-MCPPM complex (MCPPM: 4-dicyclohexylphosphino-2-diphenylphosphinomethyl-1-(*N*-methylcarbamoyl)pyrrolidine) and chiral Rh-Duanphos complex were employed to facilitate the enantioselective transformation of γ-amino ketones to chiral γ-secondary amino alcohols.[4a−c,d] In recent developments by Zhang's group, a chiral Rh−benzbisphosphine complex with $ZnCl_2$ as an activator was utilized to realize a highly efficient asymmetric hydrogenation of γ-secondary amino ketones.[4e,f,g] Despite these impressive achievements based on asymmetric hydrogenation, their practical application is still hindered by the need for sensitive chiral diphosphine ligands and high pressures of hydrogen. Therefore, exploration of more convenient asymmetric transfer hydrogenation (ATH) methods based on air-stable chiral *N*-sulfonylated diamine-based organometallic complexes,[5] especially combinations of the ATH method with aza-Michael addition[6] in a one-pot tandem process for the direct preparation of chiral γ-secondary amino alcohols from enone substrates, is of great significance in relation to the practical synthesis of chiral antidepressant drug (Scheme 1B).

Based on our recent efforts in asymmetric transfer hydrogenation,[7] we report herein the development of a convenient

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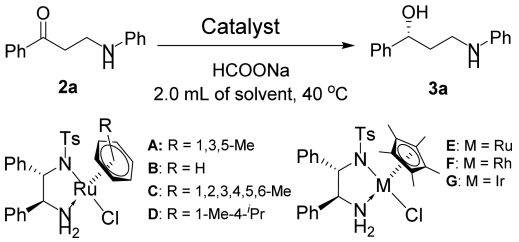
Michael addition−ATH one-pot enantioselective process for the preparation of γ-secondary amino alcohols. The superiority of this process lies in its tandem nature, and it not only offers a general synthetic approach for one-pot enantioselective organic transformations but also provides an enantioselective protocol for library screening of chiral pharmaceutical products. This meets the needs for the preparation of chiral antidepressants. As we envisaged, this Michael addition−ATH one-pot enantioselective tandem reaction of aryl-substituted enones and amines enables the production of various chiral aryl-substituted γ-secondary amino alcohols in high yields with up to 99% enantioselectivity in an environmentally friendly medium. This makes it particularly attractive for the practical synthesis of antidepressants through a sequential etherification reaction.

As one of the most important objectives in tandem asymmetric catalysis, we first optimized the enantioselectivity of the second step of ATH reduction. Due to the highly efficient aza-Michael addition of enones and amines obtained by the use of optimized reaction conditions in aqueous sodium carbonate,[8] together with the consideration of green catalysis and compatibility, we chose water as the reaction solvent and sodium formate as the hydrogen source for the optimization of second step of ATH reduction in the presence of aqueous sodium carbonate.[9] As shown in Table 1, the ATH of 1-phenyl-3-(phenylamino)propanone catalyzed by **A** afforded (*R*)-1-phenyl-3-(phenylamino)propanol in 63% yield with 77% *ee*. Due to the poor solubility of the substrate in water, which led to the poor yield, several cosolvents were screened. The results showed that all of the tested polar cosolvents significantly enhanced the yield in markedly shortened reaction times (Table 1, entry 1 versus entries 2−6). Among these cosolvents, *i*-PrOH/H_2O could provied the best enantioselectivity with up to 96% *ee* (Table 1, entry 6). Thus, *i*-PrOH/H_2O (1:1, v/v) was identified as the optimal reaction cosolvent for this catalysis system. Finally, various extensively studied η^6-arene-RuTsDpen complexes **B**−**D** (TsDpen = *N*-(*p*-toluenesulfonyl)-1,2-diphenylethylenediamine) and η^5-Cp*MTsDpen complexes **E**−**G** (η^5-Cp* = pentamethylcyclopentadiene, M = Ru, Rh, and Ir)[10] were further compared under the optimized reaction conditions (Table 1, entry 6 versus entries 7−12). Thereby, mesitylene-RuTsDpen (**A**) was identified as the optimal catalyst for the single-step of ATH enantioselective reduction. To our delight, when this ATH reduction reaction was combined with the first step of aza-Michael addition in a one-pot aza-Michael addition−ATH tandem process, catalyst **A** still afforded the target product (*R*)-1-phenyl-3-(phenylamino)propanol in 95% yield with 95% *ee* (Table 2, entry 1), which could be further confirmed by the optimization of above cosolvents in the aza-

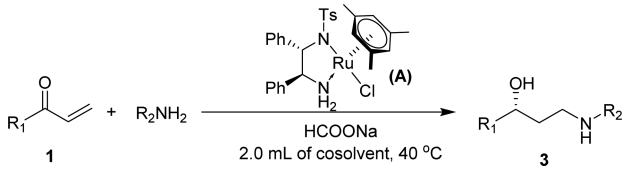
Table 1. Optimization of Solvents and Catalysts for ATH of 1-Phenyl-3-(phenylamino)propanone to (*R*)-1-Phenyl-3-(phenylamino)propanol[a]



entry	cat.	solvent	*t* (h)	yield (%)[b]	*ee* (%)[c]
1	**A**	H_2O	24	63	77
2	**A**	DMF/H_2O (1:1)	2	89	92
3	**A**	DMSO/H_2O (1:1)	2	77	91
4	**A**	EtOH/H_2O (1:1)	2	95	95
5	**A**	MeOH/H_2O (1:1)	2	80	94
6	**A**	^{i}PrOH/H_2O (1:1)	2	98	96
7	**B**	^{i}PrOH/H_2O (1:1)	24	34	84
8	**C**	^{i}PrOH/H_2O (1:1)	24	18	92
9	**D**	^{i}PrOH/H_2O (1:1)	24	45	90
10	**E**	^{i}PrOH/H_2O (1:1)	24	trace	nd
11	**F**	^{i}PrOH/H_2O (1:1)	24	57	93
12	**G**	^{i}PrOH/H_2O (1:1)	12	16	80

[a]Reactions were performed with 1.0 mol % of catalyst (1.0 μmol), 22.50 mg (0.10 mmol) of 1-phenyl-3-(phenylamino)propanone, and 20.40 mg (0.30 mmol) of HCOONa in 2.0 mL of solvent (1.0 mL of solvent and 1.0 mL of 0.10 M aqueous Na_2CO_3, 1:1, v/v) at 40 °C. [b]Isolated yield. [c]Determined by HPLC on a Daicel Chiralcel OD−H column.

Table 2. Synthesis of Chiral γ-Secondary Amino Alcohols by the aza-Michael Addition−ATH One-Pot Enantioselective Tandem Process[a]



entry	**3**	R_1, R_2	*t* (h)	yield (%)[b]	*ee* (%)[c]
1	**3a**	Ph, Ph	2	95	95
2	**3a**	Ph, Ph	2.5	93	95[d]
3	**3b**	Ph, 4-ClPh	2	83	94
4	**3c**	Ph, 3-ClPh	2	85	94
5	**3d**	Ph, 2-ClPh	2	88	94
6	**3e**	Ph, 4-BrPh	2	89	94
7	**3f**	Ph, 4-NO_2Ph	5	73	96
8	**3g**	Ph, 3-NO_2Ph	5	85	96
9	**3h**	Ph, 4-MePh	2	93	96
10	**3i**	Ph, 3,4-Me_2Ph	2	95	97
11	**3j**	Ph, 3,5-Me_2Ph	2	92	95
12	**3k**	Ph, 3-Cl-4-MePh	5	90	96
13	**3l**	Ph, 3-MeOPh	2	94	95
14	**3m**	4-FPh, Ph	5	92	90
15	**3n**	4-ClPh, Ph	5	90	92
16	**3o**	4-BrPh, Ph	5	91	92
17	**3p**	4-IPh, Ph	5	88	89
18	**3q**	4-NO_2Ph, Ph	5	86	81
19	**3r**	4-CNPh, Ph	5	82	86
20	**3s**	4-MePh, Ph	5	79	92
21	**3t**	4-MeOPh, Ph	9	80	95
22	**3u**	thienyl, Ph	4	90	98
23	**3v**	thienyl, Me	8	92	99[e]
24	**3w**	Ph, Me	10	79	96[e]
25	**3x**	Ph, Et	12	81	97[e]
26	**3y**	Ph, *n*-Bu	12	80	99[e]

[a]Reactions were performed with 1.0 μmol of **A**, 0.10 mmol of the enone, 0.11 mmol of the amine, and 0.30 mmol of HCOONa in 2.0 mL of solvent (1.0 mL of ^{i}PrOH and 1.0 mL of 0.10 M aqueous Na_2CO_3, 1:1, v/v) at 40 °C. [b]Isolated yield. [c]Determined by HPLC on Daicel Chiralcel columns. [d]Data obtained for two single-step reactions. [e]The *ee* was determined through its corresponding acetylated derivative using HPLC on Daicel Chiralcel columns.

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Michael addition−ATH one-pot tandem reaction of 1-phenylprop-2-enone and aniline (Table S1 in SI).

Having established a clean Michael addition−ATH one-pot enantioselective tandem process, we further investigated the kinetic transformation of this tandem reaction to probe the nature of the catalysis. As shown in Figure 1, it is found that the

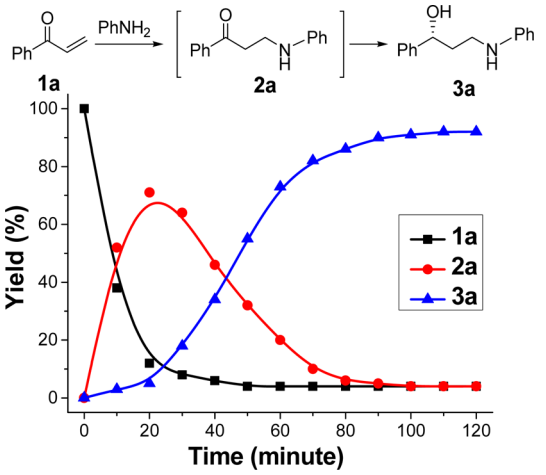


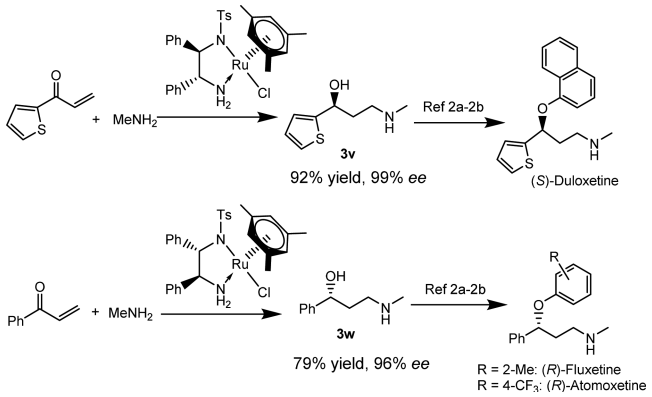
Figure 1. Time course of the aza-Michael addition−ATH of 1-phenylprop-2-enone and aniline to (*R*)-1-phenyl-3-(phenylamino)-propanol (reaction was performed with 1 equiv of 1-phenylprop-2-enone, 1.1 equiv of aniline, 1.0 mol % of **A**, 3.0 equiv of HCOONa at 40 °C).

aza-Michael addition reaction of 1-phenylprop-2-enone (**1a**) and aniline processes at first. In this stage, the concentration of **1a** decreases sharply down to 12% within 20 min, whereas 1-phenyl-3-(phenylamino)propanone (**2a**) is formed in a maximum yield of 71% with concomitant transformation of tiny (*S*)-1-phenyl-3-(phenylamino)propanol (**3a**). Subsequently, the ATH reduction of **2a** occurs quickly from 20 to 70 min. Finally, the aza-Michael addition−ATH one-pot enantioselective tandem process reaches its catalytic completion within 120 min. An advantage of this tandem process is that the enantioselective organic transformation proceeds more rapidly than can be achieved by two single-step reactions, since the latter requires 150 min to reach completion, suggesting a relatively high catalytic efficiency (Table 2, entry 1 versus entry 2).

On the basis of the obtained efficient aza-Michael addition−ATH enantioselective tandem reaction, one-pot transformations of enones and amines to various chiral aryl-substituted γ-secondary amino alcohols were further investigated to assess its applicability, as shown in Table 2. In general, high yields and enantioselectivities were obtained in all cases examined under the same reaction conditions. It was also found that the structures and electronic properties of substituents on the aromatic ring of the R_2 moiety did not significantly affect the enantioselectivity. However, slight effects on the yields were observed, with electron-withdrawing substituents on the aromatic ring of the R_2 moiety leading to relatively lower yields compared to those attained with electron-donating substituents on the aromatic ring of the R_2 moiety (entries 3−8 versus entries 9−13). In contrast, electron-withdrawing substituents on the aromatic ring of the R_1 moiety resulted in higher yields than those attained with electron-donating substituents in these positions (entries 14−19 versus entries 20−21). It was worth mentioning that the aza-Michael addition−ATH one-pot tandem reactions of 1-phenylprop-2-enone and aliphatic amines were also converted steadily (entries 23−26), which would afford the valuable intermediates for the preparation of various chiral antidepressants.[4e,g]

An important consideration in developing this aza-Michael addition−ATH tandem process was that the aim was an efficient approach for the preparation of optically pure antidepressants. As shown in Scheme 2, gram-scale syntheses of (*S*)-3-(methylamino)-1-(thiophen-2-yl)propan-1-ol and (*R*)-3-(methylamino)-1-phenylpropan-1-ol could be accomplished by tandem reactions of 1-(thiophen-2-yl)prop-2-enone and 1-phenylprop-2-enone with methanamine, followed by sequential etherification. This offers a practical method for the preparation of the antidepressants (*S*)-Duloxetine, (*R*)-Fluxetine, and (*R*)-Atomoxetine.

Scheme 2. Application of the Michael Addition−ATH Tandem Process for the Synthesis of Chiral Antidepressants



In conclusion, by employing mesitylene-RuTsDpen as a catalyst under compatible reaction conditions, we have achieved a highly efficient Michael−ATH tandem reaction for one-pot transformations of various enones to chiral γ-secondary amino alcohols. This Michael−ATH tandem process not only provides various chiral aryl-substituted γ-secondary amino alcohols in high yields and with high enantioselectivities but also offers a practical platform for the construction of various optically pure antidepressants through a sequential etherification reaction. We believe that the method described here should prove particularly attractive for the practical synthesis of pharmaceutical products.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b00823.

General information, typical experimental procedures, characterization, HPLC spectra of compound (PDF)

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Notes

The authors declare no competing financial interest.

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