

## RESEARCH ARTICLE



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## Facile synthesis of chiral indolines through asymmetric hydrogenation of *in situ* generated indoles†

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A concise and enantioselective procedure for the synthesis of optically active indolines has been developed through intramolecular condensation, deprotection and palladium-catalyzed asymmetric hydrogenation in a one-pot process with up to 96% ee. A strong Brønsted acid played an important role in both the formation of indoles and asymmetric hydrogenation process. This strategy could be scaled-up with excellent reactivity and enantioselectivity.

As a significant class of alkaloids, chiral indolines are widely prevalent in numerous bioactive compounds, such as pharmaceuticals, insecticides and herbicides.<sup>1</sup> For example, Benzastatin E has been found to be a neuronal cell protecting substance from *Streptomyces nitrosporeus* 30643 and it suppresses glutamate toxicity in N18-RE-105 cells<sup>1d</sup> (Fig. 1). To satisfy the demand for enantiomerically pure indolines, the development of efficient methods for their synthesis has attracted increasing attention.<sup>2</sup>

Asymmetric hydrogenation of indoles is one of the most straightforward approaches to afford chiral indolines with respect to operational simplicity and atom economy. However, because of the inherent aromatic stability and the poisoning effect of indoles or indolines on metal catalysts, this transformation still remains challengeable.<sup>3</sup> Since Kuwano and co-workers developed the first highly effective asymmetric hydrogenation of *N*-protected indoles catalyzed by a rhodium complex<sup>4</sup> in 2000, ruthenium<sup>5</sup> and iridium<sup>6</sup> complexes have been employed in the asymmetric hydrogenation of *N*-protected indoles, respectively. However, these transformations need to preinstall the protecting groups before the reaction and remove the protecting groups afterward. The extra steps result in the loss of yields and increase the cost in the synthetic chemistry. Subsequently, Zhou and coworkers first

reported a highly enantioselective palladium-catalyzed hydrogenation of unprotected indoles with a Brønsted acid as the activator.<sup>7</sup> Thereafter, other transition-metal catalysts<sup>8–10</sup> were involved in the asymmetric hydrogenation of *N*-unprotected indoles. Although these strategies have gained substantial progress, some of them suffer from limitations in the synthesis of the indoles and substrate scope of asymmetric hydrogenation. Therefore, it is still necessary to develop new approaches to afford chiral indolines, which remains a great challenge.

As compared with the previous synthesis of chiral indolines, asymmetric hydrogenation in a one-pot procedure<sup>11</sup> bypassing the formation of indoles is more concise, efficient and promising (Scheme 1). In this course, there was no need to isolate and purify the intermediate indoles. In addition, the byproducts were H<sub>2</sub>O, CO<sub>2</sub> and isobutylene, which was convenient for the isolation of the products. However, some challenges still remain in the process. The key point is the compatibility of the reaction conditions between formation and asymmetric hydrogenation of indoles. In addition, the stoichio-

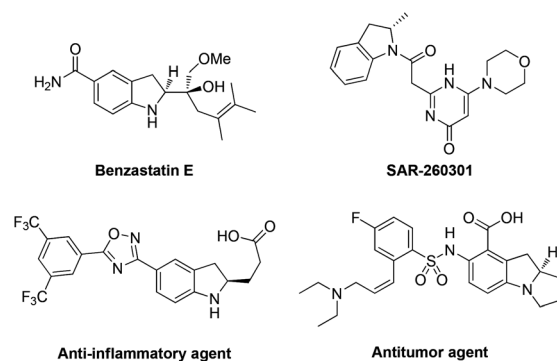


Fig. 1 Examples of chiral indoline-containing biologically active compounds.

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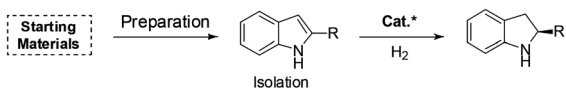
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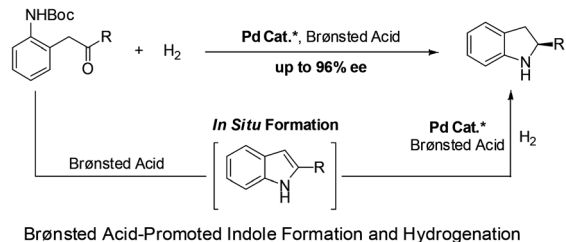
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## Previous synthesis of chiral indolines via hydrogenation of indoles



## This Work: One-pot procedure

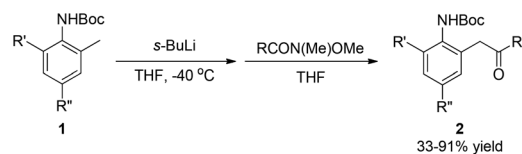


## Brønsted Acid-Promoted Indole Formation and Hydrogenation

**Scheme 1** The one-pot procedure for the synthesis of optically active indolines through the indole synthesis/asymmetric hydrogenation cascade.

metric byproduct water may poison the catalytic amount of the transition-metal catalyst. Moreover, the formation of indoles should be a fast step; otherwise, the carbonyl group may be hydrogenated directly,<sup>12</sup> leading to low yield. Herein, we report a one-pot synthesis of chiral indolines through the palladium-catalyzed asymmetric hydrogenation of *in situ* generated indoles with up to 96% ee.

Initially, the readily available *tert*-butyl (2-(2-oxo-3-phenyl-propyl)phenyl)carbamate **2a** could be synthesized from the easily accessible *N*-Boc protected *o*-toluidine **1a** and Weinreb amide in the presence of *sec*-butyllithium (Scheme 2).<sup>13</sup> Then, compound **2a** was employed as a model substrate for the investigation using the palladium-based catalytic system developed by our group.<sup>7a</sup> Fortunately, the desirable indoline **3a** was obtained with 84% ee and full conversion while using Pd(OCOCF<sub>3</sub>)<sub>2</sub>/(*S*)-SegPhos (**L1**) as the catalyst (Table 1, entry 1). Subsequently, various acids including *L*-CSA, *D*-CSA and benzoic acid were evaluated (entries 2–5). In contrast to strong acids, weak acids such as benzoic acid were ineffective. Then,



2	R	R'	R''	Yield (%)
<b>2a</b>	Bn	H	H	91
<b>2b</b>	2-MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub>	H	H	72
<b>2c</b>	3-MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub>	H	H	48
<b>2d</b>	4-MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub>	H	H	33
<b>2e</b>	Me	H	H	50
<b>2f</b>	Et	H	H	48
<b>2g</b>	<i>n</i> -Pr	H	H	35
<b>2h</b>	<i>i</i> -Pr	H	H	58
<b>2i</b>	<i>n</i> -Bu	H	H	50
<b>2j</b>	<i>n</i> -Pentyl	H	H	59
<b>2k</b>	Me	Me	H	68
<b>2l</b>	Me	MeO	H	69
<b>2m</b>	Me	H	MeO	70
<b>2n</b>	Me	Me	Me	64
<b>2o</b>	Ph	H	H	69

**Scheme 2** Synthesis of the substrates.

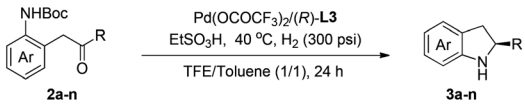
**Table 1** The evaluation of reaction parameters<sup>a</sup>

Entry	Solvent	L*	Additive	Conv. <sup>b</sup> (%)	ee <sup>c</sup>
1	TFE	<b>L1</b>	TsOH·H <sub>2</sub> O	>95	84
2	TFE	<b>L1</b>	<i>L</i> -CSA	>95	73
3	TFE	<b>L1</b>	<i>D</i> -CSA	>95	83
4	TFE	<b>L1</b>	EtSO <sub>3</sub> H	>95	86
5	TFE	<b>L1</b>	PhCO <sub>2</sub> H	—	—
6	HFIP	<b>L1</b>	EtSO <sub>3</sub> H	>95	84
7	Toluene	<b>L1</b>	EtSO <sub>3</sub> H	>95	55
8	DCM	<b>L1</b>	EtSO <sub>3</sub> H	>95	82
9	TFE	<b>L2</b>	EtSO <sub>3</sub> H	>95	88
10	TFE	<b>L3</b>	EtSO <sub>3</sub> H	>95	89
11	TFE	<b>L4</b>	EtSO <sub>3</sub> H	>95	86
12	TFE	<b>L5</b>	EtSO <sub>3</sub> H	>95	86
13	TFE	<b>L6</b>	EtSO <sub>3</sub> H	>95	80
14	TFE/Tol. (1 : 1)	<b>L3</b>	EtSO <sub>3</sub> H	>95	94
15 <sup>d</sup>	TFE/Tol. (1 : 1)	<b>L3</b>	EtSO <sub>3</sub> H	>95	95
16 <sup>d,e</sup>	TFE/Tol. (1 : 1)	<b>L3</b>	EtSO <sub>3</sub> H	>95 (98) <sup>f</sup>	95

<sup>a</sup> Conditions: **2a** (0.10 mmol), Pd(OCOCF<sub>3</sub>)<sub>2</sub>/L\* (2.5 mol%), additive (0.20 mmol), solvent (2 mL), 70 °C, H<sub>2</sub> (700 psi), 24 h. <sup>b</sup> Determined using <sup>1</sup>H NMR. <sup>c</sup> Determined using chiral HPLC. <sup>d</sup> H<sub>2</sub> (300 psi). <sup>e</sup> 40 °C. <sup>f</sup> Isolated yield at the scale of 0.25 mmol. Tol. = toluene

the solvent effect was examined and 2,2,2-trifluoroethanol (TFE) proved to be the best (entries 6–8). Further evaluations were focused on the screening of different chiral ligands (entries 9–13). However, the enantioselectivity was increased slightly from 86% ee to 89% ee while the commercially available (*R*)-H8-BINAP (**L3**) was employed. Fortunately, the mixture solvent (TFE/Tol.) brought the enantioselectivity to 94% ee (entry 14). The change of hydrogen pressure and temperature has no obvious effect on ee value (entries 15 and 16). Therefore, the optimal conditions were established: Pd(OCOCF<sub>3</sub>)<sub>2</sub>/(*R*)-**L3**/EtSO<sub>3</sub>H/TFE-Tol. (1 : 1)/H<sub>2</sub> (300 psi)/40 °C.

With the optimal conditions in hand, we set out to explore the substrate generality of the one-pot procedure for the chiral indolines through palladium-catalyzed asymmetric hydrogenation of *in situ* generated indoles. The results are summarized in Table 2. Gratifyingly, various alkyl substituted ketones performed very well under the standard reaction conditions. For 2-benzyl-substituted indoles, it was noteworthy that excellent enantioselectivities (94–95% ee) were obtained regardless of the substituents on the benzene ring (entries 1–4, **3a–d**). In addition, the length of the alkyl chain had little influence on

Table 2 Substrate scope<sup>a</sup>


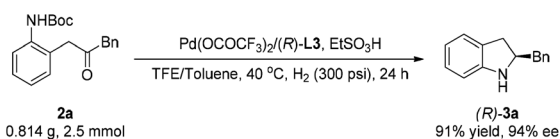
Entry	R	Ar	Yield <sup>b</sup> (%)	ee <sup>c</sup> (%)
1	Bn	H	98 (3a)	95
2	2-MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub>	H	91 (3b)	94
3	3-MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub>	H	90 (3c)	95
4	4-MeC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub>	H	93 (3d)	95
5	Me	H	96 (3e)	90
6	Et	H	82 (3f)	94
7	<i>n</i> -Pr	H	84 (3g)	94
8	<i>i</i> -Pr	H	94 (3h)	96
9	<i>n</i> -Bu	H	98 (3i)	94
10	<i>n</i> -Pentyl	H	97 (3j)	93
11	Me	2-Me	94 (3k)	96
12	Me	2-MeO	91 (3l)	80
13	Me	4-MeO	81 (3m)	84
14	Me	2,4-Me <sub>2</sub>	90 (3n)	94
15	Ph	H	55 (3o)	68

<sup>a</sup> Conditions: **2** (0.25 mmol), Pd(OCOCF<sub>3</sub>)<sub>2</sub>/(*R*)-L3 (2.5 mol%), EtSO<sub>3</sub>H (0.50 mmol), TFE/Tol. (2 mL/2 mL), 40 °C, H<sub>2</sub> (300 psi), 24 h. <sup>b</sup> Isolated yields. <sup>c</sup> Determined using chiral HPLC.

the ee values, and the high steric hindrance of the isopropyl group rarely affected the reaction activity and enantioselectivity (entries 5–10, **3e–j**, 90–96% ee). As for the methyl or methoxy substituted aryl, the reaction also proceeded smoothly, providing the chiral indolines with high enantioselectivities (entries 11–14, **3k–n**, 80–96% ee). For aryl substituted ketone **2o**, moderate yield and enantioselectivity were obtained under the above standard conditions. This phenomenon indicated that the isomerization from enamine to iminium was relatively difficult when the aryl substituent was introduced on the 2-position.<sup>9</sup>

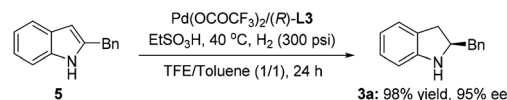
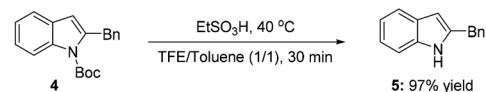
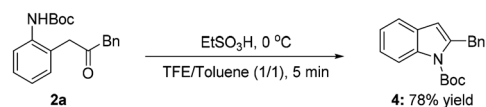
Moreover, to further demonstrate the practicality of this strategy, the scale-up experiment was carried out at 2.5 mmol scale (Scheme 3). To our delight, the desired chiral indoline **3a** was obtained without the loss of activity and enantioselectivity (91% yield, 94% ee).

To obtain further insight into the reaction pathway, several control experiments were carried out, as summarized in Scheme 4. Treatment of **2a** with ethanesulfonic acid led to the *N*-Boc protected indole **4** with 78% yield in five minutes at 0 °C. When the *N*-Boc protected indole **4** was reacted with ethanesulfonic acid at 40 °C, it transformed into 2-benzyl-1*H*-indole **5** quickly with 97% yield. The results showed that the formation of indole was a fast step. Then, the isolated indole **5** was reacted under the standard conditions, giving the same

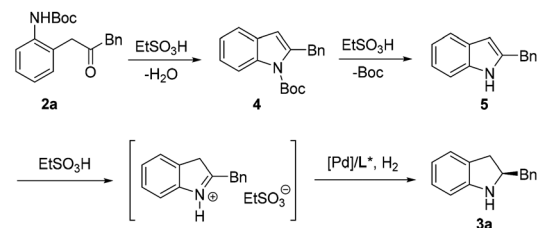


Scheme 3 The scale-up experiment.

## The Control Experiments:



## The Proposed Reaction Pathway:



Scheme 4 The mechanism investigation.

chiral indoline **3a** with full conversion and an identical enantioselectivity. These experimental results supported the reaction pathway that the compound **2a** converted into indole **5** through sequential intramolecular condensation and *N*-Boc deprotection quickly in the presence of a strong Brønsted acid. Then, the intermediate indole **5** was activated by a Brønsted acid to form an iminium intermediate *in situ*, followed by asymmetric hydrogenation to afford the final product.<sup>14</sup>

## Conclusion

In summary, we have developed a highly efficient and enantioselective procedure for the synthesis of optically active indolines through intramolecular condensation, *N*-Boc deprotection and palladium-catalyzed asymmetric hydrogenation in a one-pot process. By this route, *tert*-butyl (2-(2-oxo-3-phenylpropyl)phenyl) carbamates could be conveniently transformed into the corresponding chiral 2-substituted indolines with excellent yields and up to 96% ee. A strong Brønsted acid played an important role in both the formation of indoles and asymmetric hydrogenation process. In addition, this strategy could be scaled up with excellent reactivity and enantioselectivity. Further investigations of cascade reactions involving asymmetric hydrogenation for the synthesis of chiral amines are undergoing in our laboratory.

## Experimental

## A one-pot procedure for the synthesis of chiral indolines

Ligand (*R*)-H8-BINAP (4.8 mg, 0.0076 mmol) and Pd(OCOCF<sub>3</sub>)<sub>2</sub> (2.1 mg, 0.0063 mmol) were placed in a dried Schlenk tube

under a nitrogen atmosphere, and degassed anhydrous acetone was added. The mixture was stirred at room temperature for one hour. The solvent was removed under vacuum to give the catalyst. This catalyst was taken into a glove box filled with nitrogen and dissolved in 2,2,2-trifluoroethanol (2 mL). To the mixture of compound **2a** (0.25 mmol) and ethanesulfonic acid (0.50 mmol, 41  $\mu$ L) in toluene (2 mL), this catalyst solution was added, and then the mixture was transferred to an autoclave, which was charged with hydrogen gas (300 psi). The autoclave was stirred at 40 °C for 24 h. After careful release of the hydrogen, the autoclave was opened and the reaction mixture was evaporated. Then, saturated sodium bicarbonate (5 mL) was added. The mixture was extracted with dichloromethane (5 mL  $\times$  3), and the combined organic layer was dried over sodium sulfate and concentrated *in vacuo*. The residue was purified using flash chromatography using ethyl acetate/hexanes (10/1) as the eluent to give the chiral products **3a** as a colorless oil (51 mg, 98% yield, 95% ee). Enantiomeric excess was determined using HPLC (OD-H column, *n*-hexane/*i*-PrOH 99/1, 1.0 mL min<sup>-1</sup>, 254 nm, 30 °C),  $t_1$  = 14.8 min (major),  $t_2$  = 16.6 min (minor).

## Conflicts of interest

There are no conflicts to declare.

## Acknowledgements

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