



# Enantioselective and divergent construction of chiral amino alcohols and oxazolidin-2-ones *via* Ir-*f*-phamidol-catalyzed dynamic kinetic asymmetric hydrogenation

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## ABSTRACT

The dynamic kinetic resolution (DKR) process remains a highly efficacious approach for constructing chiral amino alcohols *via* the catalytic asymmetric hydrogenation of  $\alpha$ -amino ketones. We report herein a highly efficient and enantioselective anti-selective dynamic kinetic asymmetric hydrogenation of  $\alpha$ -amino ketones catalyzed by Ir-(*S*)-*f*-phamidol system, providing various chiral amino alcohols and chiral oxazolidin-2-ones divergently with high diastereo- and enantioselectivity (up to 99% yield, up to 99% *ee* and up to 99:1 *dr*). In addition, the reaction could be performed on the gram-scale, and the resulting chiral amino alcohols are key intermediates of norephedrine and metamaminol.

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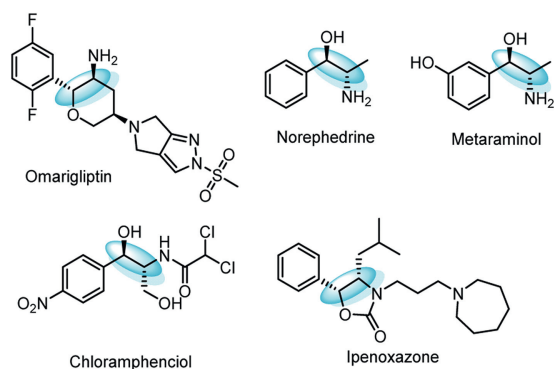
Chiral  $\alpha$ -amino alcohol fragments are a crucial constituent of pharmacologically active molecules and drug compounds [1–5]. They serve as pivotal intermediates in various chiral drug pharmacophores, including chloramphenicol [6], metamaminol [7], norephedrine, and omargliptin [8,9] as outlined in Fig. 1. Notably, chloramphenicol exhibits potent antibacterial activity against Gram-negative bacteria [10], while metamaminol is frequently utilized as a rescue medication for patients experiencing shock. Furthermore, this molecular fragment is extensively utilized as a chiral ligand in the domain of asymmetric synthesis owing to the nitrogen atom's strong coordination ability in the amino group [11,12]. In addition to their potential bioactivity [13,14], chiral 2-oxazolidones are commonly used as chiral auxiliaries in organic synthesis [15–17]. Given the significance of chiral  $\alpha$ -amino alcohols and chiral 2-oxazolidones, the pursuit of a succinct, cost-effective, and effective approach for synthesizing chiral  $\alpha$ -amino alcohols and chiral 2-oxazolidones is a prominent area of investigation in the field of green synthetic chemistry. One of the most effective and straightforward methods to construct chiral amino alcohols is the asymmetric hydrogenation of  $\alpha$ -amino ketones.

With the development of asymmetric catalysis, dynamic kinetic resolution (DKR) has proved to be one of the most powerful and influential strategies for introducing novel stereogenic centers into the synthesis of pharmaceutical ingredients and natural products [18]. Since 1989, Noyori's group has reported pioneering work on the utilization of Ru(BINAP)Cl<sub>2</sub> (Noyori's 1<sup>st</sup> Gen catalyst) as the catalyst for the enantioselective hydrogenation of racemic  $\alpha$ -acetamido  $\beta$ -keto esters *via* DKR process [19]. This seminal work has significantly invigorated the wide range of DKR process of transition-metal-catalyzed organic reactions [20–24]. In 2004, our group disclosed a highly enantio- and diastereoselective asymmetric hydrogenation of  $\alpha$ -phthalimide ketones to form  $\alpha$ -phthalimide alcohols *via* DKR [25], which was achieved using the Ru/C<sub>3</sub>-TunePhos as the catalyst, resulting in the production of *syn*- $\alpha$ -phthalimide alcohols with high conversion and enantioselectivity (Scheme 1A).

With the discovery and development of Noyori's 2<sup>nd</sup> Gen catalyst and the popularity of outer sphere mechanism in ketone asymmetric hydrogenation [26–29], various ruthenium [30–33] and iridium [34–36] catalysts have been developed to catalyze the dynamic kinetic asymmetric hydrogenation of  $\alpha$ -amino ketones. In 2009, Zhou and coworkers reported a highly enantioselective asymmetric hydrogenation of *N*-disubstituted amino ketones with excellent enantio- and diastereoselectivity utilizing Ru(SDP)(diamine) catalyst [31]. In 2011, a novel iridium catalyst

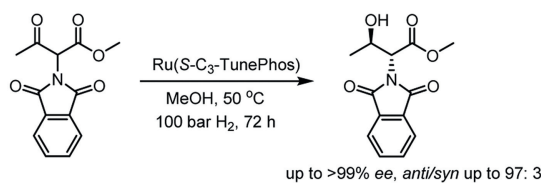
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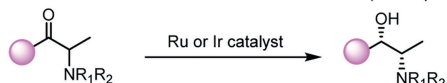


**Fig. 1.** Key pharmacologically active compounds containing chiral amino alcohol or oxazolidin-2-one moiety.

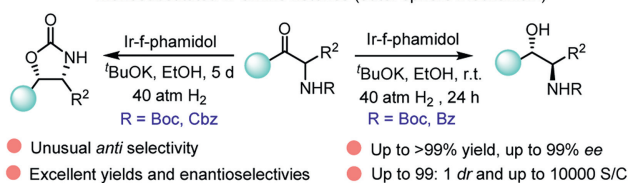
(A) Ruthenium catalyzed asymmetric hydrogenation (inner sphere mechanism)



(B) *Syn*-selective asymmetric hydrogenation of *N*-disubstituted  $\alpha$ -amino ketones (outer sphere mechanism)



(C) **This work:** *Anti*-selective divergent asymmetric hydrogenation of *N*-monosubstituted  $\alpha$ -amino ketones (outer sphere mechanism)

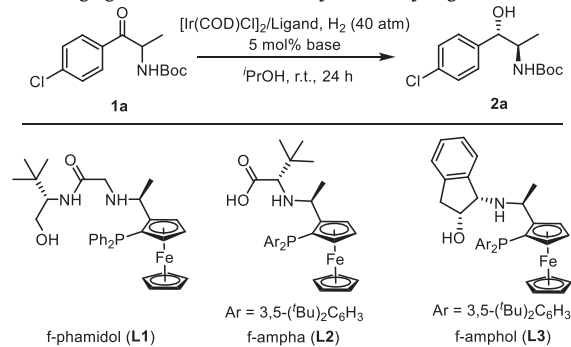


**Scheme 1.** Ruthenium catalyzed asymmetric hydrogenation (A). Iridium catalyzed asymmetric hydrogenation (B) and this work (C) on symmetric reduction of the *N*-heteroaryl ketones.

bearing tridentate SpiroPAP ligand [37–39] was developed by Zhou and coworker which showed high reactivity and enantioselectivity in the asymmetric hydrogenation of  $\alpha$ -amino ketones [40] and lactones [41]. Since 2016, a series of tridentate ligands skeleton were reported by our group. These ligands, namely f-amphox [34,35,42–45], f-ampha [46–48], f-amphol [49–51], f-amphamide [52], and f-phamidol [36,45], were found to be exhibited high catalytic activity upon complexation with iridium. Furthermore, they were capable of catalyzing the asymmetric hydrogenation of various ketone substrates with high yield (>99%) and excellent enantioselectivity (>99% *ee*). The Ir-f-amphox system has the ability to catalyze the asymmetric hydrogenation of racemic  $\alpha$ -amino  $\beta$ -unfunctionalized ketones *via* DKR process to construct chiral  $\alpha$ -amino  $\beta$ -unfunctionalized alcohols with >99% yield, >99% *ee*, >99:1 *dr* and TON up to 100,000 (Scheme 1B) [34]. Additionally, this DKR process facilitates the preparation of crucial chiral intermediates for preclinical antitumor drug (S,S)-R116010. In 2022, our group reported the utilization of the Ir-f-phamidol system for the asymmetric hydrogenation of aryl  $\alpha$ -dibenzylamino  $\beta$ -ketoesters and  $\alpha$ -dibenzylamino aromatic ketones, resulting in the successful synthesis of *syn*- $\beta$ -hydroxy  $\alpha$ -amino acid derivatives with excellent diastereo- and enantioselectivities (Scheme 1B) [36]. In addition to asymmetric hydrogenation, ruthenium-catalyzed asym-

**Table 1**

Screening ligands and bases for the asymmetric hydrogenation of **1a**.<sup>a</sup>



Entry	Ligand	Base	Yield (%) <sup>b</sup>	<i>dr</i> <sup>c</sup>	<i>ee</i> (%) <sup>c</sup>
1	L1	<sup>t</sup> BuOK	>99	96/4	>99
2	L2	<sup>t</sup> BuOK	9	50/50	15
3	L3	<sup>t</sup> BuOK	13	54/46	22
4	L1	Cs <sub>2</sub> CO <sub>3</sub>	>99	75.5/24.5	>98
5	L1	K <sub>2</sub> CO <sub>3</sub>	19	56/44	87
6	L1	NaOH	>99	95/5	>99
7	L1	LiOH	44	92.5/7.5	95
8	L1	<sup>t</sup> BuONa	>99	78.5/21.5	97
9	L1	<sup>t</sup> BuOLi	>99	95/5	98

<sup>a</sup> Reaction conditions: **1a** (0.10 mmol), S/C = 500, base (0.005 mmol), <sup>t</sup>PrOH (1.0 mL), r.t., 40 atm H<sub>2</sub>.

<sup>b</sup> Determined from the results of NMR analysis.

<sup>c</sup> Determined from the results of HPLC analysis on a chiral stationary phase.

metric transfer hydrogenation of  $\alpha$ -amino ketones was also reported by Somfai [53,54] and Wills's group [55]. Despite great advances in the asymmetric hydrogenation of  $\alpha$ -amino ketones, the current research mainly focused on the asymmetric hydrogenation of *N*-disubstituted  $\alpha$ -amino ketones, with limited attention given to the asymmetric hydrogenation of *N*-monosubstituted  $\alpha$ -amino ketones [35]. Herein, we disclose a highly practical and efficient iridium(I)-catalyzed enantioselective and divergent hydrogenation of *N*-monosubstituted  $\alpha$ -amino ketones *via* DKR with high yield and excellent chemo-, diastereo- and enantioselectivity (up to 99% yield and up to 99% *ee*), and a series of synthetically important chiral amino alcohols and oxazolidin-2-ones were provided efficiently (Scheme 1C).

In our initial investigation, we employed *tert*-butyl(1-(4-chlorophenyl)-1-oxopropan-2-yl)carbamate (**1a**) as a model substrate in <sup>t</sup>PrOH with <sup>t</sup>BuOK as base and [Ir(COD)Cl]<sub>2</sub> as transition metal precursor under 40 atm H<sub>2</sub> at room temperature. To explore the impact of ligands on the asymmetric hydrogenation of **1a**, we evaluated three distinct ligands, f-amphol (**L3**), f-ampha (**L2**), and f-phamidol (**L1**) (Table 1, entries 1–3), the iridium complex of which has demonstrated high reactivity and enantioselectivity in the asymmetric hydrogenation of simple ketones. Regrettably, the other two catalysts using **L2** and **L3** as the ligand failed to effectively facilitate the hydrogenation of **1a**, and a very low conversion (9%–13%) and poor enantio- and diastereoselectivity were observed (Table 1, entries 2 and 3). To our delight, the catalyst, Ir-f-phamidol, could fully achieve conversion to obtain the product of **2a**, with ideal 96:4 *dr* and >99% *ee* (Table 1, entry 1).

Given the significant impact of alkalinity on catalytic activity and selectivity, we conducted the experiments using various bases, such as Cs<sub>2</sub>CO<sub>3</sub>, K<sub>2</sub>CO<sub>3</sub>, NaOH, LiOH, <sup>t</sup>BuONa and <sup>t</sup>BuOLi (Table 1, entries 4–9), in comparison with <sup>t</sup>BuOK. Our findings indicate that the use of K<sub>2</sub>CO<sub>3</sub> or LiOH at S/C = 500 in the reaction system resulted in low conversions and reaction rates (Table 1, entries 5 and 7). Although other bases yielded ideal conversions, they failed to achieve the better *dr* and *ee*, thereby falling short of the cat-

**Table 2**  
Screening solvents for the asymmetric hydrogenation of **1a**.<sup>a</sup>

Entry	Solvent	Yield (%) <sup>b</sup>	<i>dr</i> <sup>c</sup>	<i>ee</i> (%) <sup>c</sup>
1	<i>i</i> PrOH	99 ( <b>2a</b> )	96/4	>99
2	MeOH	10 ( <b>2a</b> )	90/10	79
3	EtOH	99 ( <b>2a</b> )	98.5/1.5	>99
4	THF	36 ( <b>2a</b> )	93.5/6.5	96
5	EtOAc	10 ( <b>2a</b> )	96.5/3.5	>99
6	DCM	9 ( <b>2a</b> )	94/6	90
7 <sup>d</sup>	EtOH	99 ( <b>2a</b> )	98/2	>99
8 <sup>e</sup>	EtOH	99 ( <b>2a</b> )	98.9/1.1	>99
9 <sup>f</sup>	EtOH	95 ( <b>3a</b> )	98/2	>99

<sup>a</sup> Reaction conditions: **1a** (0.10 mmol), S/C = 500, <sup>t</sup>BuOK (0.005 mmol), solvent (1.0 mL), r.t., 40 atm H<sub>2</sub>, 24 h.

<sup>b</sup> Determined from the results of NMR analysis.

<sup>c</sup> Determined from the results of HPLC analysis on a chiral stationary phase.

<sup>d</sup> <sup>t</sup>BuOK (0.11 mmol), EtOH (1.0 mL), r.t., 40 atm H<sub>2</sub>, 24 h.

<sup>e</sup> S/C = 10,000, <sup>t</sup>BuOK (0.11 mmol), EtOH (1.0 mL), r.t., 40 atm H<sub>2</sub>, 24 h.

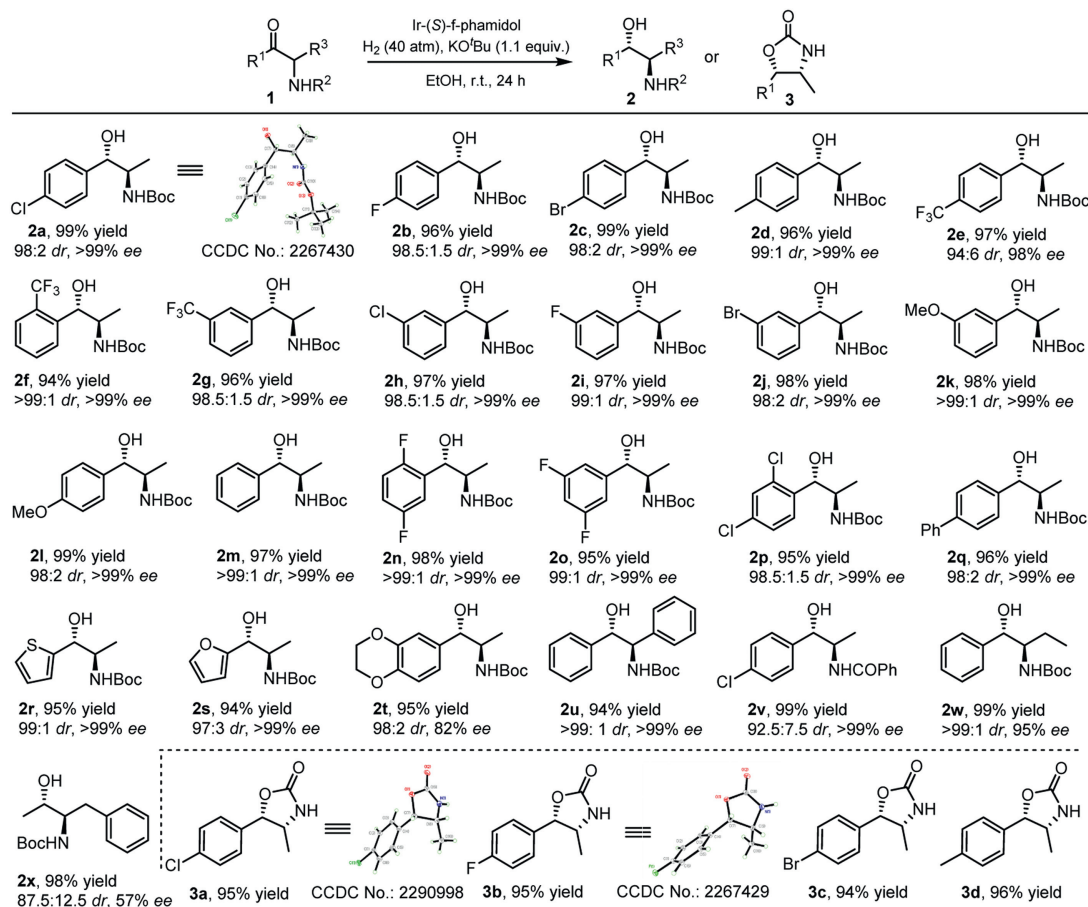
<sup>f</sup> 1.1 equiv. of <sup>t</sup>BuOK was added, 5 d.

alytic efficacy of <sup>t</sup>BuOK in the reaction system. Therefore, <sup>t</sup>BuOK was found to be the best choice for the reaction system.

After the screening of ligands and bases, the influence of solvents on the reaction was also investigated at S/C = 500, and the results were depicted in Table 2. Using MeOH as solvent instead

of *i*PrOH, the yield was dropped to 10%, and 90:10 *dr* and 79% *ee* were observed (Table 2, entry 2). To our delight, the reaction in EtOH proceeded smoothly to provide **2a** with 99% yield, 98.5:1.5 *dr* and >99% *ee* (Table 2, entry 3). Aprotic solvents such as THF, DCM and EtOAc were also evaluated, only 9%–36% yield was achieved and the enantioselectivity and diastereoselectivity were not better than that of EtOH (Table 2, entries 4–6). The reaction proceeded smoothly when the amount of base was increased to 1.1 equiv., and the reaction also worked well at a substrate/catalyst ratio of 10,000 with retention of enantio- and diastereoselectivity (Table 2, entries 7 and 8). It should be noted that, chiral oxazolidin-2-one **3a** was obtained with 95% yield in the presence of 1.1 equiv. <sup>t</sup>BuOK after 5 days (Table 2, entry 9). Based on the aforementioned screening results, we identified the optimal conditions for this reaction Ir-f-phamidol complex as the catalyst, <sup>t</sup>BuOK as the base and EtOH as the solvent with a substrate concentration of 0.1 mol/L under 40 atm of H<sub>2</sub> at room temperature for 24 h. The absolute configuration of the product **2a** was unambiguously determined by the X-ray diffraction of its single crystal. The relative configuration of **2a** was found to be *anti*, which is different from the result of asymmetric hydrogenation of *N*-disubstituted  $\alpha$ -amino ketones previously reported by our group [34,56].

With the optimal reaction conditions in hand, we next put our efforts to examining the substrate scope of the reaction, and the results were listed in Scheme 2. Regrettably, some substrates, such as **1b** and **1c**, exhibited low conversions, probably due to the acidic nature of “NH” moiety in the substrate, which poses challenges in achieving optimal catalytic effects with catalytic amount of base. Pleasingly, the reaction proceeded smoothly



**Scheme 2.** Substrate scope of the reaction. Reaction conditions: **1** (0.10 mmol), S/C = 500, <sup>t</sup>BuOK (0.11 mmol), EtOH (1.0 mL), r.t., 40 atm H<sub>2</sub>, 24 h; conversion was detected by <sup>1</sup>H NMR of crude product; isolated yield of product, the *dr* and *ee* values were determined by chiral HPLC analysis.

when the amount of base was increased to 1.1 equiv. The impact on conversion and stereoselectivities due to alterations in the parent benzene rings was assessed. For the substrates containing electron-withdrawing group such as bromo, chloro, fluoro and trifluoromethyl substituents on the *ortho*-, *meta*-, or *para*-position of the phenyl ring (**1a–1c** and **1e–1j**), a consistently excellent yields, enantio- and diastereoselectivities (94:6 to >99:1 *dr*, and 98% to >99% *ee*) were exhibited. Similarly, compounds bearing two chloro or fluoro groups on the phenyl rings, **1n**, **1o**, **1p**, could also achieve the excellent catalytic results (>98.5:1.5 *dr* and >99% *ee*). The substrates with electron-donating groups, namely methyl, methoxy and phenyl (**1d**, **1k**, **1l**, **1m** and **1q**), were also investigated and excellent enantio- and diastereoselectivity (>98:2 *dr* and >99% *ee*) were observed. It is worth noting that substrates **1k** and **1m**, the asymmetric hydrogenation products of which, have the potential to be transformed into the key intermediate of metaraminol and norephedrine following the reported procedure [57–59]. Heteroaromatic substrates containing thiophene or furan moiety (**1r** and **1s**) were also subjected to the asymmetric reduction conditions. substrates **1r** and **1s** displayed high yields (94%–95% yields) and excellent enantioselectivities (>97:3 *dr* and >99% *ee*), whereas, only 82% *ee* was obtained for substrate **1t**, although it still maintained excellent enantioselectivity (98:2 *dr*). Moreover, the substitution of the methyl group with a phenyl (**1u**) or ethyl (**1w**) group, as well as the utilization of other *N*-protecting groups (**1v**), were tested to assess the reaction's efficiency. Notably, all variations exhibited a noteworthy level of *ee* and *dr* values, achieving greater than 95% *ee* and 92.5:7.5 *dr* in all cases. The dialkyl substrate **1x** demonstrated a high yield of 98%, while only moderate enantioselectivity of 57% was achieved. Considering the importance of chiral oxazolidin-2-ones in organic synthesis, the substrate scope for the formation of cyclic chiral oxazolidin-2-ones were also tested, and chiral oxazolidin-2-ones bearing a chloro, fluoro, bromo or methyl group on the *para* position of the phenyl group (**3a–3d**) could be efficiently produced with 94% to 96% yield in the presence of 1.1 equiv. of *t*BuOK by extending the reaction time to 5 days. It should be noted that the reaction of substrate with *N*-Cbz group (**1y**) proceeded smoothly to provide the cyclic product **3a** within 24 h (for details see Supporting information). The X-ray crystallographic analysis provided unequivocal assignment of the absolute configurations of product **2a**, **3a** and **3b**, demonstrating the attainment of identical *anti*-configuration (see Supporting information for details). By analogy, it is hypothesized that the remaining asymmetric hydrogenation products **2** and **3** exhibit the same trend.

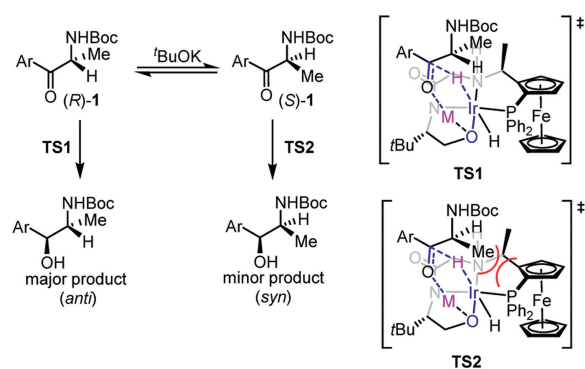
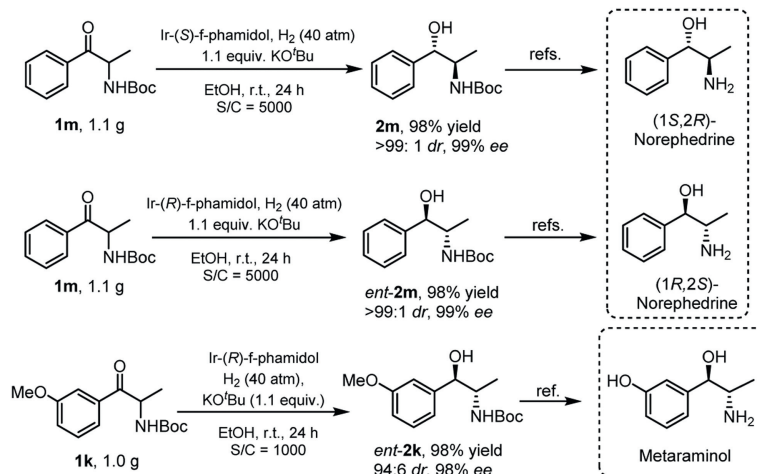


Fig. 2. Proposed models to elucidate enantiomeric induction in Ir-(S)-f-phamidol catalyzed hydrogenation of **1** via DKR.

To elucidate the pronounced stereoselectivity exhibited by the Ir-(S)-f-phamidol catalyst, a model was proposed to account for the origin of diastereoselectivity, based on our previous experimental and theoretical investigation on Ir-f-phamidol-catalyzed asymmetric hydrogenation of acetophenone [45,61]. As can be seen from Fig. 2, in the presence of *t*BuOK, (*S*)-**1** and (*R*)-**1** are in equilibrium with each other, the transition state **TS1** leading to *anti* product is favorable in energy compared with **TS2**, because the unfavorable steric interaction between the methyl group of (*S*)-**1** and the PPh<sub>2</sub> group of the ligand **L1** in **TS2** will increase its energy.

To demonstrate the potential synthetic applications of the current methodology, we performed gram-scale asymmetric hydrogenation with *S/C*=5000 and *S/C*=1000 respectively, and the results were depicted in Scheme 3. Under optimized asymmetric hydrogenation conditions, with Ir-(S)-f-phamidol or Ir-(R)-f-phamidol as catalyst and EtOH as solvent, the reaction of **1m** proceeded smoothly on 1.1 g-scale, providing the desired product *anti*-*tert*-butyl ((1*S*,2*R*)-1-(4-chlorophenyl)-1-hydroxypropan-2-yl)carbamate (**2m**) and *anti*-*tert*-butyl ((1*R*,2*S*)-1-(4-chlorophenyl)-1-hydroxypropan-2-yl)carbamate (*ent*-**2m**) with 98% isolated yield, 99% *ee* and >99:1 *dr* within 24 h (Scheme 3). According to the reported procedure, compound **2m** (*ent*-**2m**) could be transformed into norephedrine in one step. In addition, the gram-scale asymmetric hydrogenation of **1k** using Ir-(R)-f-phamidol as the catalyst also proceeded smoothly at *S/C*=1000 on 1.0 g scale, affording the corresponding product *tert*-butyl ((1*R*,2*S*)-1-hydroxy-1-(3-methoxyphenyl)propan-2-



Scheme 3. Scale-up reactions and synthetic applications.

yl)carbamate (*ent*-**2k**), with 98% yield and 98% *ee* within 24 h (Scheme 3).

In brief, we have demonstrated the remarkable catalytic efficacy of Ir(*S*)-*f*-phamidol in the asymmetric hydrogenation of *N*-monosubstituted  $\alpha$ -amino ketones *via* DKR under mild reaction conditions, resulting in divergent and *anti*-selective formation of chiral  $\alpha$ -amino alcohols and chiral oxazolidin-2-ones in nearly perfect enantioselectivity and diastereoselectivity (up to >99% *ee* and up to >99:1 *dr*). Notably, this reaction proceeded smoothly at room temperature. The synthetic potential of the current reaction has been validated by the successful gram-scale asymmetric hydrogenation of compound **1k** and **1m**, the product of which can be transformed to norephedrine and metamaminol feasibly. Further potential synthetic applications of this methodology in asymmetric synthesis of chiral drugs are ongoing in our lab, and the results will be reported in due course.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### CRediT authorship contribution statement

**Ruixue Liu:** Writing – original draft, Methodology, Investigation. **Xiaobing Ding:** Validation, Formal analysis. **Qiwei Lang:** Project administration. **Gen-Qiang Chen:** Writing – review & editing, Project administration, Conceptualization. **Xumu Zhang:** Project administration.

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### Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ccl.2024.110037.

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