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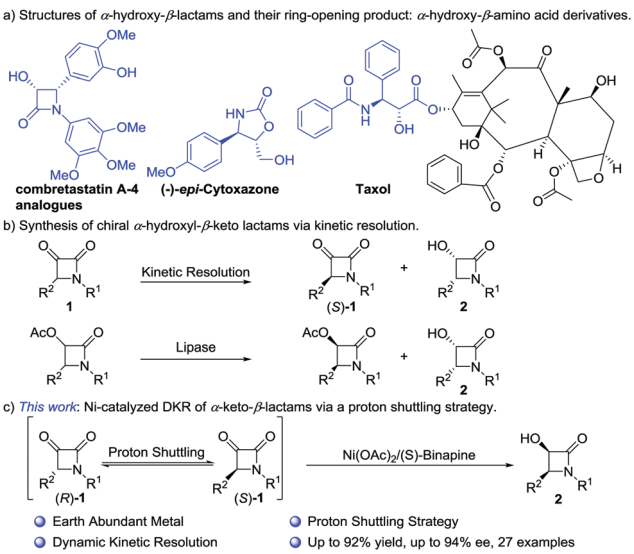
Ni-Catalyzed asymmetric reduction of α-keto-β-lactams *via* DKR enabled by proton shuttling†

Fangyuan Wang,‡[ab] Xuefeng Tan,‡[b] Ting Wu,[c] Long-Sheng Zheng,[b] Gen-Qiang Chen *[d] and Xumu Zhang *[b]

Chiral α-hydroxy-β-lactams are key fragments of many bioactive compounds and antibiotics, and the development of efficient synthetic methods for these compounds is of great value. The highly enantioselective dynamic kinetic resolution (DKR) of α-keto-β-lactams was realized *via* a novel proton shuttling strategy. A wide range of α-keto-β-lactams were reduced efficiently and enantioselectively by Ni-catalyzed asymmetric hydrogenation, providing the corresponding α-hydroxy-β-lactam derivatives with high yields and enantioselectivities (up to 92% yield, up to 94% ee). Deuterium-labelling experiments indicate that phenylphosphinic acid plays a pivotal role in the DKR of α-keto-β-lactams by promoting the enolization process. The synthetic potential of this protocol was demonstrated by its application in the synthesis of a key intermediate of Taxol and (+)-*epi*-Cytoxazone.

Chiral β-lactams (2-azetidinone) are widely found in many bioactive compounds, especially in antibiotics.[1] Ever since the first synthesis of β-lactams in 1907 by Staudinger,[2] β-lactam compounds have attracted the attention of many organic chemists during the last few decades.[3] What's more, chiral α-hydroxy-β-lactams are the key fragment of many pharmaceutically active molecules such as glycosides,[4] anti-microbial drugs[5] and anti-cancer reagents[6] (Scheme 1a). Chiral α-hydroxyl-β-amino acid derivatives,[7] the ring-opening products of α-hydroxy-β-lactams, are ubiquitously found in many medicinal targets and bioactive compounds,[8] for example, the side chain of Taxol[9] and (−)-*epi*-cytoxazone[10] (Scheme 1a). Thus, the efficient synthesis of chiral α-hydroxy-β-lactams is of great value. The asymmetric hydrogenation of α-keto-β-lactams is one of the most straightforward approaches to access these compounds and the corresponding α-keto-β-lactams can be easily synthesized *via* [2+2] cycloaddition between imines and *in situ* generated ketenes (Staudinger reaction).[11]

Dynamic kinetic resolution (DKR) is an exceedingly useful tool in asymmetric catalysis because it allows the full conversion of a racemic reactant into a chiral product.[12] This is in sharp contrast to the standard kinetic resolution, which necessarily has a maximum yield of 50%, and rapid interconversion between the two enantiomers of the reactant was necessary to achieve effective DKR.[13] The DKR of carbonyl compounds has been a well-established protocol in asymmetric catalysis, and the addition of a strong base is usually necessary to promote the enolization process.[14] Due to their low stability under basic



Scheme 1 (a) Examples of biologically active compounds derived from α-hydroxy-β-lactams. (b) Previous work. (c) This work.

[a] School of Chemistry and Chemical Engineering, Harbin Institute of Technology, Harbin 150001, People's Republic of China
[b] Shenzhen Grubbs Institute and Department of Chemistry, Guangdong Provincial Key Laboratory of Catalysis, Southern University of Science and Technology, Shenzhen, 518055, China. E-mail: zhangxm@sustech.edu.cn
[c] College of Innovation and Entrepreneurship, Southern University of Science and Technology, 1088 Xueyuan Road, Shenzhen, 518055, China
[d] Academy for Advanced Interdisciplinary Studies, Southern University of Science and Technology, Shenzhen 518000, People's Republic of China. E-mail: chengq@sustech.edu.cn

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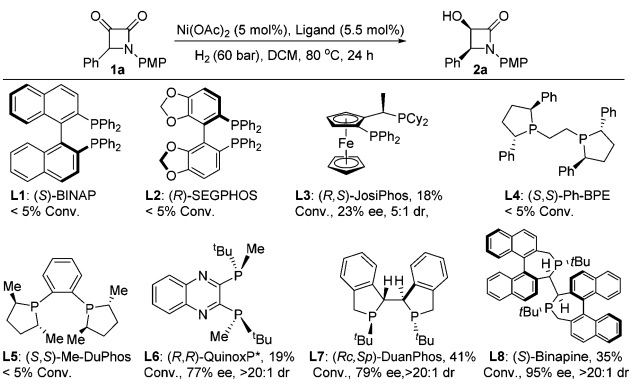
‡ These authors contributed equally to this work.

conditions, the DKR of α-keto-β-lactams is rarely reported, and α-keto-β-lactams are usually reduced *via* chemical or enzymatic kinetic resolutions (Scheme 1b).[15] In 2005, Kayser and co-workers reported an example of enzymatic DKR with high enantioselectivity, albeit with low diastereoselectivity and narrow substrate scope.[16] To date, the dynamic kinetic reduction of α-keto-β-lactams is still a significant challenge, and the key to realizing the DKR is to accelerate the enolization process (a proton transfer process). In 2019, Dong and co-workers realized the DKR of aldehyde by hydroacylation, and an amine was utilized to promote the enolization–racemization process *via* an enamine intermediate,[17] which inspired us to explore some additives that are rarely used in the DKR process. We envisioned that interconversion between the two enantiomers ((*R*)-**1** and (*S*)-**1**) of α-keto-β-lactams **1** could be promoted by an acidic proton shuttling catalyst.[18] Herein, we report the highly efficient and enantioselective dynamic kinetic asymmetric hydrogenation of α-keto-β-lactams, wherein, a novel proton shuttling strategy was utilized to promote the enolization–racemization process (Scheme 1c).

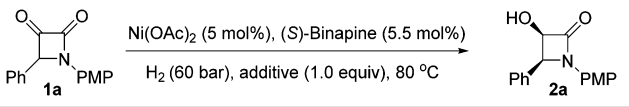
We initiated our investigation with the asymmetric hydrogenation of compound **1a** by the protocols previously developed in our group. The enantioselectivity of product **2a** was very low for the Ir(I) catalytic system,[19] the Ru(II)/diphosphine system[20] and the transfer hydrogenation system[21] (for details see Tables S1–S3 in the ESI†), probably due to the fact that the interconversion between the two enantiomers was very slow and the two enantiomers of **1a** were both rapidly reduced. Thus, we focused our attention on the less reactive earth-abundant metal Ni(II).[22] The efficacy of several diphosphine ligands was evaluated in the Ni-catalyzed asymmetric reduction using DCM as a solvent, and the results are depicted in Scheme 2. When BINAP (**L1**) or SEGPHOS (**L2**) was used as a ligand, the conversion was very low even though the temperature was raised to 80 °C. 18% conversion, 23% ee and 5 : 1 dr were achieved with (*R*,*S*)-JosiPhos (**L3**). Electron-donating ligands such as (*S*,*S*)-Ph-BPE (**L4**) and (*S*,*S*)-Me-DuPhos (**L5**) were not suitable for the current reaction, and <5% conversion was observed in both cases. To our great delight, with (*R*, *R*)-Quinoxp* (**L6**) as a ligand, 19% conversion and 77% ee were observed. The conversion and enantioselectivity were improved to 41% and 79% respectively with (*Rc*,*Sp*)-DuanPhos (**L7**) as a ligand. The enantioselectivity could be further increased to 95% using (*S*)-Binapine (**L8**)[23] as a ligand, albeit with only 35% conversion.



Scheme 2 Evaluation of the effect of various ligands.

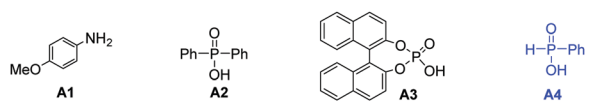
Encouraged by these promising results, we further screened various solvents, temperatures and additives, and the results are summarized in Table 1. When the temperature was decreased from 80 °C to 65 °C (entry 1), only 17% conversion was observed. 48% conversion and 95% ee were achieved at 75 °C after 48 h (entry 2). The conversion was increased to 58% with the retention of enantioselectivity when the reaction was conducted at 100 °C for 24 h, while the ee value dropped sharply after further extension of the reaction time (entries 3–5). Various solvents such as THF, toluene, hexane and methanol were also screened at 80 °C, and no improvement in conversion and enantioselectivity was observed (entries 6–9). Next, we focused our attention on the evaluation of the effect of additives on the current reaction. In the presence of K_2CO_3, the reaction became messy (entry 10). 84% conversion and 45% ee were achieved when NaBARF was added (entry 11). 8% and 45% ee were observed for the additive TsOH and AcOH, respectively (entries 12 and 13). No desired product was detected when *p*-methoxy aniline (**A1**) was utilized as an additive (entry 14).[17] 75% conversion and 65% ee were achieved for the additive diphenylphosphinic acid (**A2**) (entry 15). Only 7% conversion and 25% ee were achieved for phosphoric acid (**A3**) (entry 16). To our great delight, product **2a** was produced in >95% conversion and 81% ee with phenylphosphinic acid (**A4**)

Table 1 Optimization of the reaction conditions[a]



Entry	Solvent	Additive	*t* (h)	Conv.[b] (%)	dr[c]	ee[d] (%)
1[e]	DCM	—	36	17	>20 : 1	90
2[f]	DCM	—	48	48	>20 : 1	95
3[g]	DCM	—	24	58	>20 : 1	95
4[g]	DCM	—	36	90	>20 : 1	37
5[g]	DCM	—	72	>95	>20 : 1	12
6	THF	—	24	35	>20 : 1	93
7	Toluene	—	24	23	>20 : 1	96
8	Hexane	—	24	<5	—	—
9	MeOH	—	24	Messy	—	—
10	DCM	K_2CO_3	24	Messy	—	—
11	DCM	NaBArF	24	84	3 : 1	45
12	DCM	TsOH	24	>95	>20 : 1	8
13	DCM	AcOH	24	56	>20 : 1	45
14	DCM	**A1**	24	—	—	—
15	DCM	**A2**	24	75	>20 : 1	65
16	DCM	**A3**	24	7	>20 : 1	25
17	DCM	**A4**	24	>95	>20 : 1	81
18	**Toluene**	**A4**	**36**	**>95**	**>20 : 1**	**91**

[a] All reactions were carried out on 0.1 mmol scale. [b] Determined *via* ^{1}H NMR spectroscopy. [c] Determined *via* ^{1}H NMR spectroscopy. [d] The ees were determined by HPLC analysis using a chiral stationary phase. [e] 65 °C. [f] 75 °C. [g] 100 °C.



as an additive (entry 17). The enantioselectivity was further improved to 91% when toluene was used as a solvent (entry 18).

With the optimal reaction conditions in hand, we investigated the substrate scope of the current reaction. First, the effect of *N*-protecting groups on the current reaction was evaluated and the results are summarized in Scheme 3. For substrates with electron-donating or electron-withdrawing groups on the phenyl ring of the *N*-protecting groups (**2b–2i**), high yields and moderate to high enantioselectivities were achieved (81% to 90% yield, 84% to 91% ee). The reaction also worked well for substrates with benzyl or alkyl *N*-protecting groups (**2j–2m**, 68% to 77% yield, 88% to 94% ee). In addition, the effect of substituents on the γ-position was also evaluated. With PMP as the *N*-protecting group, 87% yield and 91% ee were achieved (**2a**), which were comparable with the results of substrates **2b** and **2j**. PMP was selected as the standard *N*-protecting group because it can be easily removed in the subsequent step. For substrates with an electron-donating group (**2n–2o**), an electron-withdrawing group (**2p**) or halogens (**2q–2s**) on the para position of the phenyl ring, the reaction proceeded smoothly to deliver the desired products in high yields and enantioselectivities (81% to 91% yield, 87% to 94% ee). Moderate yield and enantioselectivity can be achieved for the substrate with an amide group on the para position of the phenyl ring (**2t**, 45% yield, 78% ee). The reaction also tolerates substrates with *meta*-(**2u**, 85% yield, 87% ee) or *ortho*-substituents (**2v**, 80% yield, 82% ee) on the phenyl ring. 92% yield and 88% ee were achieved for 2-naphthyl substituted substrate **2w**. The current reaction also worked well for thienyl and furyl substituted substrates (**2x** and **2y**, 85% and 88% yield, 82% and 87% ee, respectively) and alkyl substrates (**2z** and **2aa**, 70% and 91% yield, 85% and 90% ee, respectively). The diastereoselectivities were high for all the substrates tested (**1a–1aa**, $>20:1$ dr). The main limitation of this reaction is that low yield was generally achieved for substrates with electron-withdrawing groups on the phenyl group, in the case of **1t**, a considerable amount of the ring-opening product was detected. The substrate scope of the current reaction is also restricted by the fact that the synthesis of substrates with electron-withdrawing groups on the phenyl group and with normal alkyl groups on the β position was problematic.

A probable mechanism for the dynamic kinetic resolution was proposed and depicted in Scheme S2 in the ESI.† After the introduction of phenylphosphinic acid (**A4**), (*R*)-**1a** could be transformed to the enol intermediate **1a′** *via* an eight-membered transition state, and the enol intermediate **1a′** could isomerize to compound (*S*)-**1a**, which was reduced much faster than its enantiomer (*R*)-**1a**. Deuterium-labelling experiments were also conducted to elucidate the role of the additive **A4** (for details see Scheme S2 in the ESI†).

To demonstrate the synthetic potential of the current methodology, the reduction of **1a** was conducted on 1.0 mmol scale, and product **2a** was obtained in 85% yield and 91% ee under standard conditions. After protection with TIPSCl, oxidative removal of the PMP group and acylation with benzoyl bromide, the side chain of Taxol could be efficiently synthesized (Scheme 4a).[9] The reduction of **1n** could also be conducted on 1.0 mmol scale, and product **2n** was produced in 91% yield and 92% ee. In the presence of TMSCl and MeOH, compound **5n**, the key intermediate of (+)-*epi*-cytoxazone, was synthesized in 90% yield and 92% ee (Scheme 4b).[10]

In conclusion, we have developed a Ni-catalyzed highly efficient methodology for the dynamic kinetic asymmetric reduction of α-keto-β-lactams, providing the α-keto-β-lactam products in high enantioselectivity and diastereoselectivity. The dynamic kinetic resolution (DKR) of α-keto-β-lactams is realized *via* a novel proton shuttling strategy. Deuterium-labelling experiments indicate that phenylphosphinic acid plays a pivotal role in the enolization of α-keto-β-lactams. Further applications of this strategy in the DKR of carbonyl compounds are underway in our group and the results will be reported in due course.

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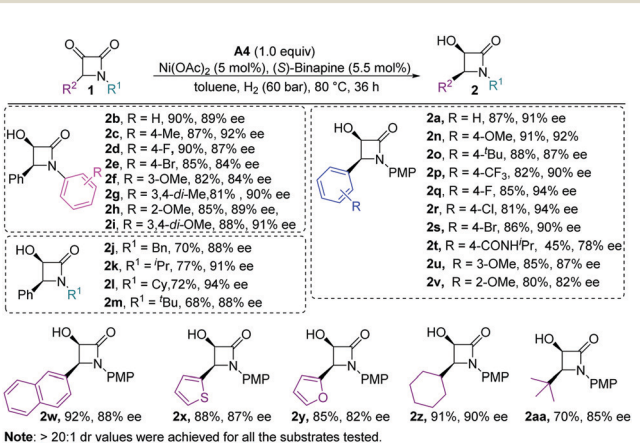
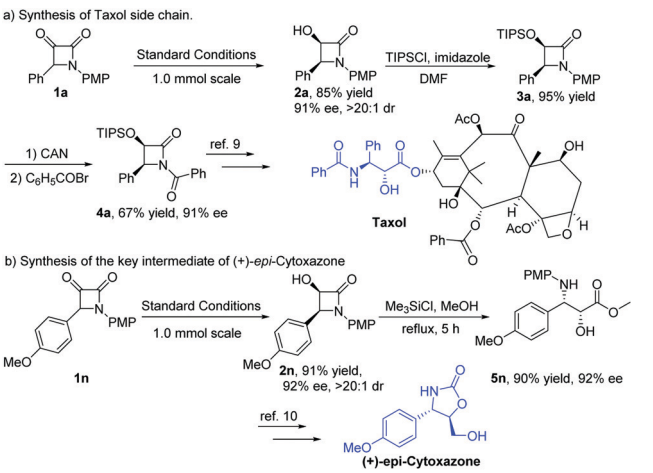


Scheme 3 Evaluation of the substrate scope.



Scheme 4 Synthetic elaborations of the Ni-catalyzed DKR.

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Conflicts of interest

There are no conflicts to declare.

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