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Organocatalytic asymmetric synthesis of oxazolines from *N*-acylimines

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ABSTRACT

A novel organocatalytic asymmetric approach to oxazoline derivatives that proceeds through Mannich/annulation reaction of *N*-acylimines with 3-chlorooxindoles is presented. This strategy provides an efficient and convenient method to access enantioenriched oxazolines such as valuable chiral *S,N*-oxazoline ligand as well as Ferrox ligand in high yields with excellent enantio– and diastereroselectivity. Furthermore, the optically active oxazoline products can be converted to valuable 1,2-amino alcohols. More importantly, the synthetic utility of this transformation is demonstrated in the expeditious assembly of chiral Phox-type ligand, which shows excellent catalytic activities.

The oxazoline structural motifs are valuable intermediates in organic transformations, glycosyl hydrolysis and polymer chemistry [1–8], and they are also prevalent scaffolds in numerous natural products and bioactive molecules [9–14]. Chiral oxazoline motifs play an important role in asymmetric synthesis, extensively serving as a preeminent class of chiral ligands (Fig. 1) [15–23]. Accordingly, the extensive use of enantioenriched oxazoline scaffolds has spawned widespread interest in the development of efficient enantioselective synthesis of optically active oxazolines, and great efforts have been devoted to this field. Based on the existing oxazole or oxazoline skeleton, chiral oxazolines can be synthesized by catalytic asymmetric hydrogenation of oxazoles [24] or alkylation of oxazolines [25–27]. In addition, catalytic asymmetric intramolecular cyclization of *N*-allylamides [28–39] or *N*-propargylamides [40], Heine reaction of *N*-acylaziridines [41], and desymmetrization of aminodiols [42–44] have been developed for enantioselective synthesis of oxazolines. Generally, the most common and well-established asymmetric approach to construct oxazolines is the aldol reaction of isocyanoacetates with carbonyl compounds (Scheme 1a). Since the first asymmetric aldol reaction of isocyanoacetates with aldehydes catalyzed by chiral gold(I) catalyst was reported in 1986 by Ito and Hayashi [45], a number of transition-metal complexes, including gold [46–48], silver [49–53], palladium [54–56], platinum [57,56,58,59], and cobalt [60] have been demonstrated to be effective for this aldol-type condensation. Moreover, organocatalysts [61–65] as well as the combination of transition-metal catalysis and organocatalysis [66,67], have emerged as efficient catalyst systems in this reaction for accessing chiral oxazolines in the last decade. Recently, Denmark reported another type of intermolecular asymmetric synthesis of oxazolines from alkenes and *N*-tosylbenzamides (Scheme 1b) [68]. This chiral diselenide catalyzed enantioselective alkene 1,2-oxyamination proceeded in high yields with excellent enantioselectivities. Despite these notable advancements, the development of new catalytic asymmetric intermolecular methods for the facile construction of enantiomerically pure oxazolines from a diverse array of starting materials is still highly desirable.

In sharp contrast to the abovementioned well-developed formal [3 + 2] annulation strategies, catalytic asymmetric formal [4 + 1] annulation strategy for the assembly of oxazolines is unexplored. There are some challenging issues for this [4 + 1] transformation. First is the identification of suitable four-atom starting materials that can be activated by chiral organocatalysts. Second is the identification of reactive C1 reaction partners bearing a suitable dual nucleophilic/electrophilic character that can be activated by chiral organocatalysts to undergo asymmetric [4 + 1] annulation. The final issue is the identification of effective chiral organocatalysts to control the reactivity, diastereoselectivity, and enantioselectivity of the formal [4 + 1] annulation. In this context, we envisioned that *N*-acylimines as four-atom reaction partners and 3-chlorooxindoles as C1 reaction partners might be suitable to access chiral oxazolines through enantioselective Mannich/annulation reaction pathway. This consideration is based on four aspects. Firstly, *N*-acylimines are highly reactive electrophiles. The presence

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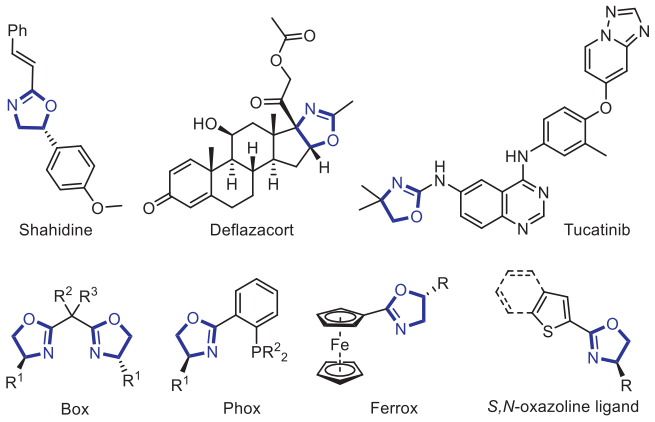
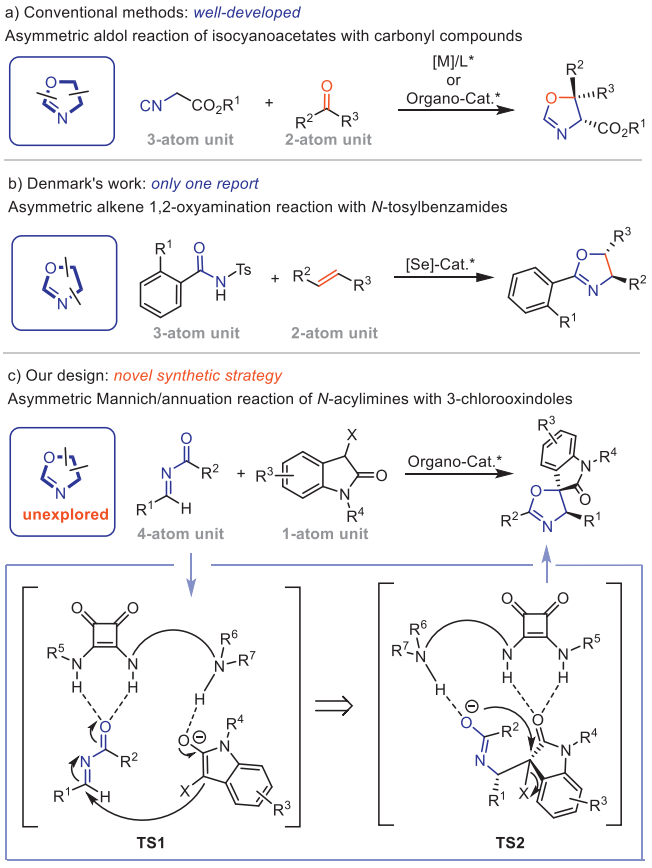


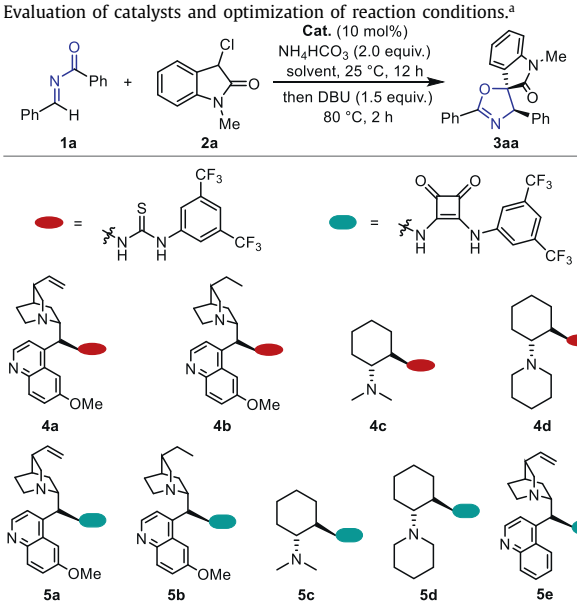
Fig. 1. Oxazolines in bioactive compounds, natural products, and ligands.



Scheme 1. Catalytic enantioselective synthesis of oxazolines.

of electron-withdrawing *N*-acyl moiety imparts to the azomethine carbon a particularly high level of electrophilicity. Secondly, because of the presence of acyl group, after the nucleophilic addition, the obtained amino intermediates are amenable in the subsequent ring closure operation. Thirdly, *N*-acylimines can be activated by chiral organocatalysts. Therefore, *N*-acylimines are particularly appreciated as suitable four-atom substrates. Fourthly, the introduction of chloro group at the C-3 position of indoles serves as an excellent leaving group in the subsequent annulation. In addition, this also increases the acidity of the C–H bond at the C-3 position of indoles. Inspired by the dual nucleophilic/electrophilic reactivity profile, it is assumed that 3-chlorooxindoles could serve as C1 reaction partners. Herein, we reported a novel and efficient organocatalytic enantioselective formal [4+1] annulation of *N*-

Table 1
Evaluation of catalysts and optimization of reaction conditions.[a]

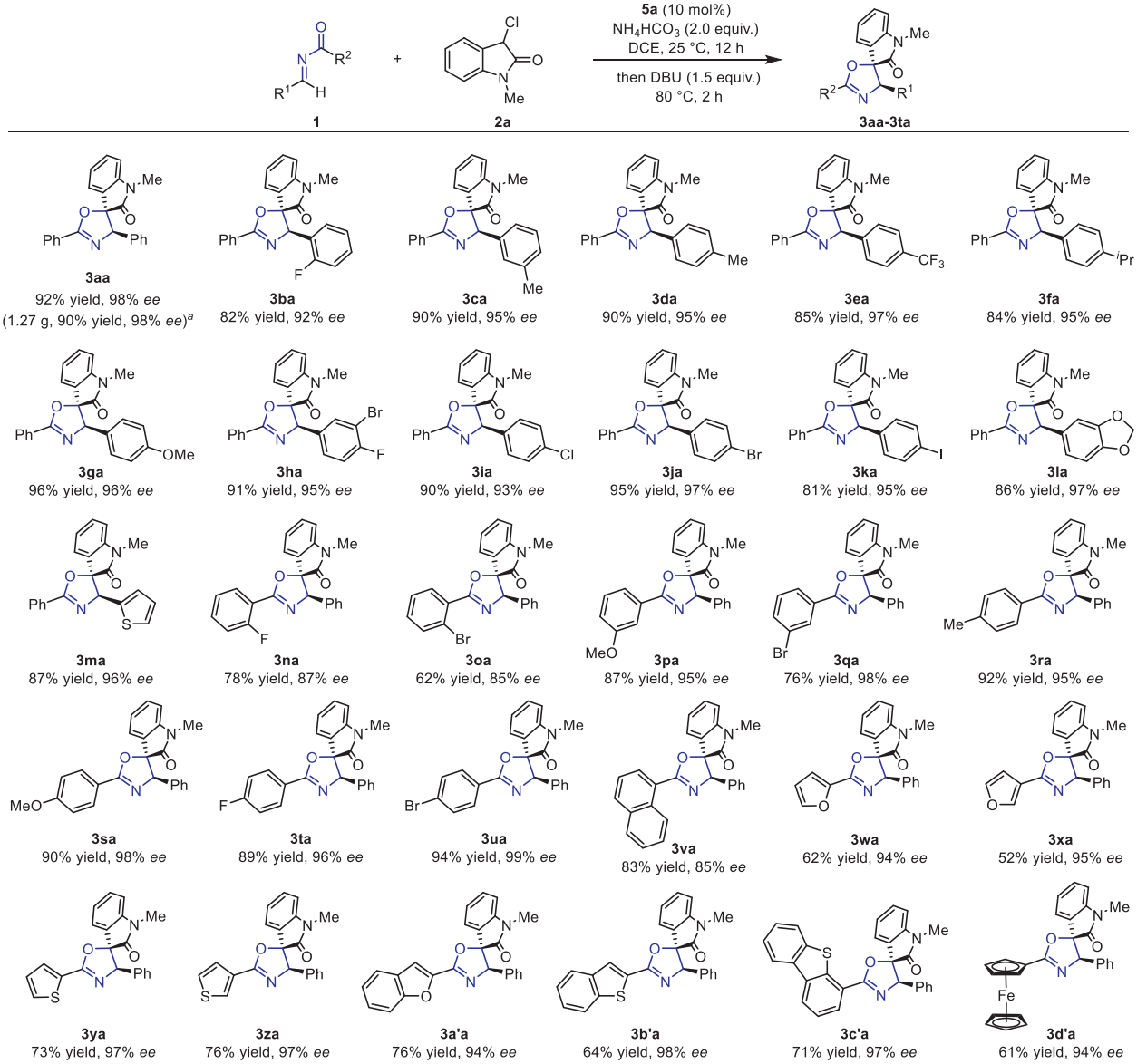


Entry	Cat.	Solvent	Yield (%)[b]	*ee* (%)[c]
1	**4a**	DCE	86	91
2	**4b**	DCE	81	90
3	**4c**	DCE	76	−34
4	**4d**	DCE	73	−55
5	**5a**	DCE	92	98
6	**5b**	DCE	90	97
7	**5c**	DCE	<5	–
8	**5d**	DCE	<5	–
9	**5e**	DCE	78	85
10	**5a**	DCM	87	97
11	**5a**	$CHCl_3$	85	91
12	**5a**	EA	73	93
13	**5a**	THF	82	81
14	**5a**	1,4-Dioxane	75	95
15	**5a**	Toluene	81	75

[a] Reaction conditions: **1a** (0.10 mmol), **2a** (0.15 mmol), NH_4HCO_3 (0.20 mmol) and catalyst (0.01 mmol) in solvent (1.0 mL) at 25 °C for 12 h. In the final annulation step, DBU (0.15 mmol) was used.
[b] Isolated yield, and the *dr* value was >19:1 in all cases.
[c] The *ee* value was determined by HPLC.

acylimines with 3-chlorooxindoles for synthesizing a diverse range of oxazolines. The plausible transition state models that account for the bifunctional activation mode are presented in Scheme 1c. Both *N*-acylimine and 3-chlorooxindole are activated by the chiral organocatalyst through hydrogen-bonding interaction, which facilitates the stereoselective Mannich addition/annulation sequence, realizing the asymmetric [4+1] annulation.

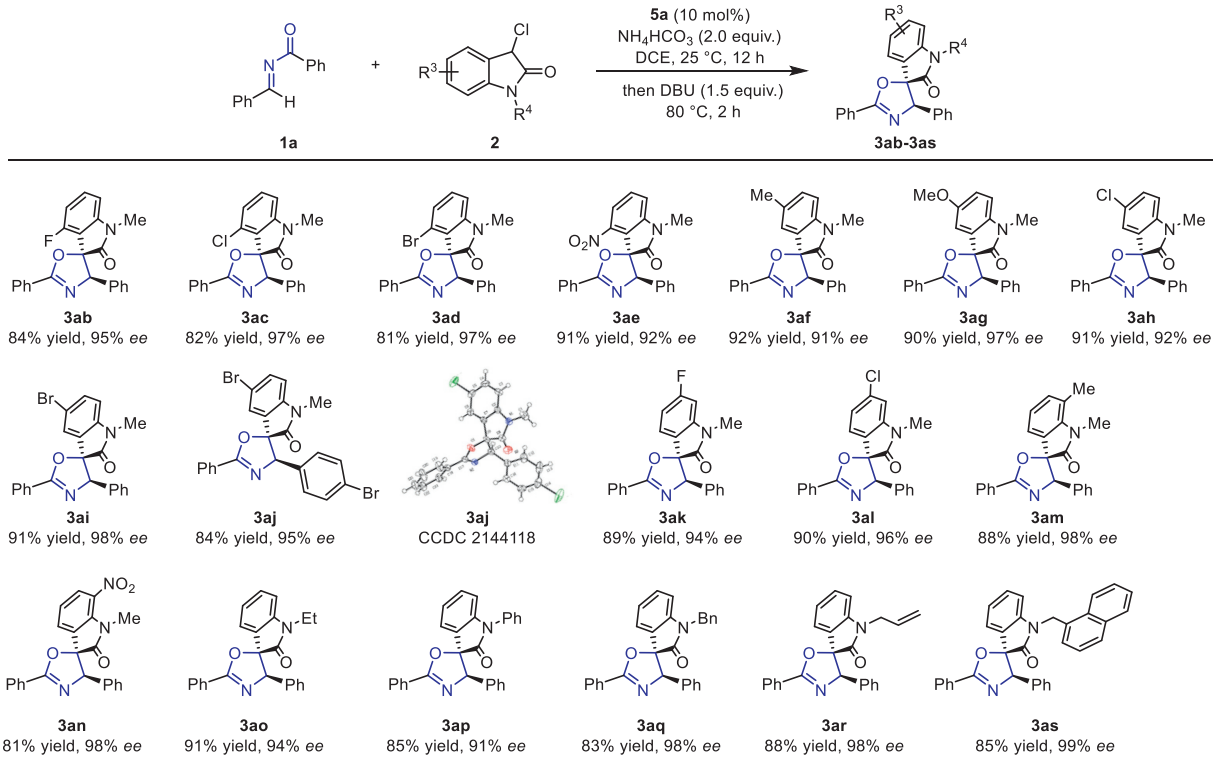
With this design, the reaction of *N*-acylimine **1a** with 3-chlorooxindole **2a** was investigated initially as a model reaction to test the designed formal [4+1] annulation, with the key results summarized in Table 1. The reaction was first conducted in 1,2-dichloroethane at 25 °C, then DBU was used for the final annulation. Gratifyingly, in the presence of chiral cinchona alkaloid-derived thiourea organocatalyst **4a**, a smooth [4+1] cyclization reaction indeed occurred, affording the oxazoline product **3aa** in 86% yield with an excellent diastereoselectivity of >19:1 *dr*, and high enantioselectivity of 91% *ee* (entry 1). This preliminary result demonstrated the feasibility of our hypothesis. In order to improve the enantioselectivity, a series of chiral bifunctional tertiary amine catalysts were screened (entries 2–9), and it was found that chiral cinchona alkaloid-derived squaramide catalyst **5a** performed the best in enantioselectivity control (entry 5). Then, **5a** was selected as the optimal catalyst to examine the solvent effect (en-



Scheme 2. Substrate scope of *N*-acylimines. Reaction conditions: **1** (0.10 mmol), **2a** (0.15 mmol), NH_4HCO_3 (0.20 mmol) and **5a** (0.01 mmol) in DCE (1.0 mL) at 25 °C for 12 h. In the final annulation step, DBU (0.15 mmol) was used. Isolated yield. The *dr* value was >19:1 in all cases. The *ee* value was determined by HPLC. [a] 4 mmol scale of **1**.

tries 10–15). Finally, the optimal conditions were identified to be the following: **5a** as catalyst and 1,2-dichloroethane as solvent with NH_4HCO_3 as base at 25 °C for 12 h, delivering **3aa** in 92% yield with excellent *dr* (>19:1) and *ee* (98%, entry 5).

With the optimal reaction conditions in hand, large-scale reaction on 4 mmol of *N*-acylimine under the standard condition delivered oxazoline **3aa** in 90% yield with no loss of the enantioselectivity (98% *ee*), demonstrating the robustness of this asymmetric [4 + 1] annulation. Consequently, a series of *N*-acylimines with 3-chlorooxindoles were selected to examine the tolerance of substitution patterns and functional groups in this enantioselective formal [4 + 1] annulation. This methodology was shown to be efficient for a variety of *N*-acylimines (Scheme 2). All of the evaluated aromatic *N*-acylimines bearing electron-donating and electron-withdrawing groups could provide the desired chiral oxazoline products **3ba**-**3ka** in high yields with excellent diastereoselectivities and enantioselectivities, regardless of substitution patterns on the aromatic ring. The imines with benzo[*d*][1,3]dioxol-5-yl and 2-thienyl substitution also gave good yields (86% yield for **3la**, 87% for **3ma**) and high enantioselectivities (97% *ee* for **3la**, 96% *ee* for **3ma**). It is noted that aliphatic imines failed to provide the targeted oxazolines. Various alkyl-substituted imines, such as methyl, *tert*–butyl and cyclohexyl were tested, however decomposition results were obtained. This might be due to the isomerization of aliphatic imines to the corresponding enamines. In addition, aliphatic imines are more unstable and susceptible to hydrolysis compared to aromatic imines. The scope of the reaction with respect to variation of acyl part of *N*-acylimines was then explored. Electron-rich, electron-poor and halide substitution provided access to oxazolines in excellent yields and enantioselectivities. *Ortho*-, *meta*- and *para*-substitution and naphthyl-substitution on the aryl ring were tolerated well (**3na-3va**), demonstrating good tolerance to steric crowding. Reactions of heteroatom-substituted acyl part took place smoothly, leading to the corresponding oxazolines **3wa-3c'a** in high yields and excellent enantioselectivities. It is noteworthy that *S,N*-oxazoline ligands containing oxazoline functionality and thiophene, benzothiophene or dibenzothiophene as an auxiliary donor ligands [69,70] could be obtained through this



Scheme 3. Substrate scope of 3-chlorooxindoles. Reaction conditions: **1a** (0.10 mmol), **2** (0.15 mmol), NH_4HCO_3 (0.20 mmol) and **5a** (0.01 mmol) in DCE (1.0 mL) at 25 °C for 12 h. In the final annulation step, DBU (0.15 mmol) was used. Isolated yield. The *dr* value was >19:1 in all cases. The *ee* value was determined by HPLC.

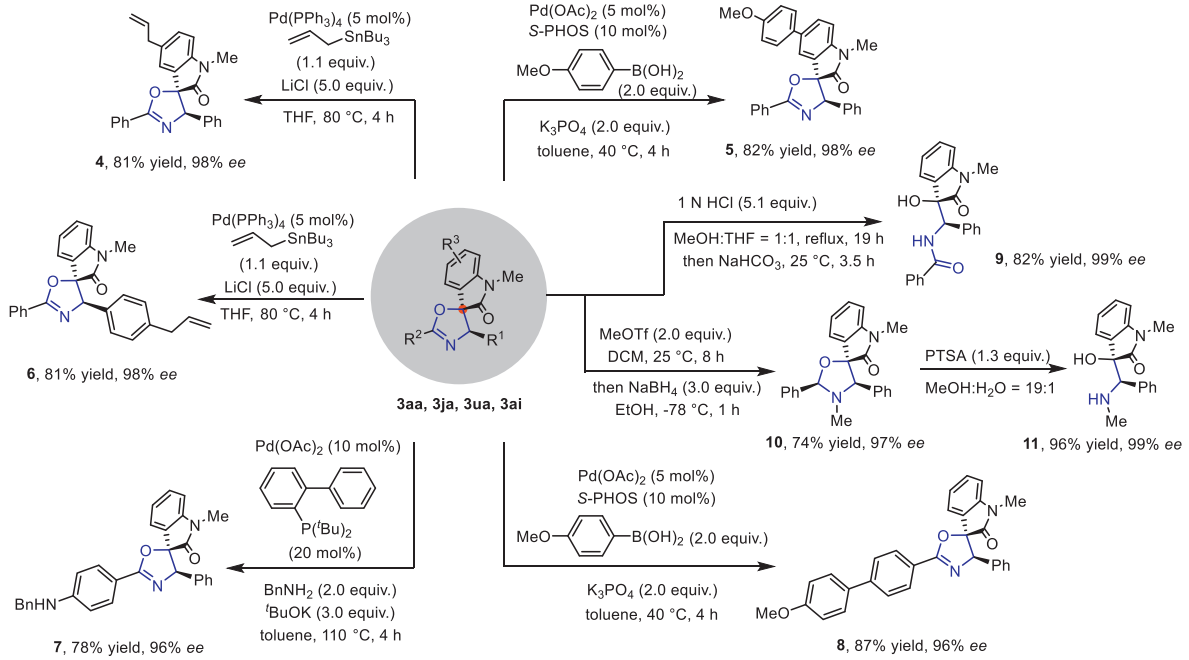
method. Moreover, the R2 group of *N*-acylimines could be ferrocenyl, furnishing chiral ferrocenyl-oxazoline **3d'a**, a type of new Ferrox ligand [71,72], in 61% yield with 94% *ee*.

The broad reaction scope of this formal [4 + 1] annulation was further demonstrated with different 3-chlorooxindoles (Scheme 3). The reaction tolerated broad variation in the substituent electronics of the phenyl ring on 3-chlorooxindoles, irrespective of their positions on the aromatic ring, delivering enantioenriched oxazoline products with high yields and enantioselectivities. Increasing the steric crowding and electron-withdrawing property of functional groups on the aromatic ring were not detrimental to the reaction. The reaction was also tolerant for different *N*-substituents on the 3-chlorooxindoles, and various *N*-substituted 3-chlorooxindoles were found to be amenable to the reaction conditions, giving the corresponding products **3ao**-**3as** in excellent yields and enantioselectivities. The absolute configuration of the chiral oxazoline products was determined by the single crystal X-ray diffraction of **3aj**, and those of other products were assigned accordingly.

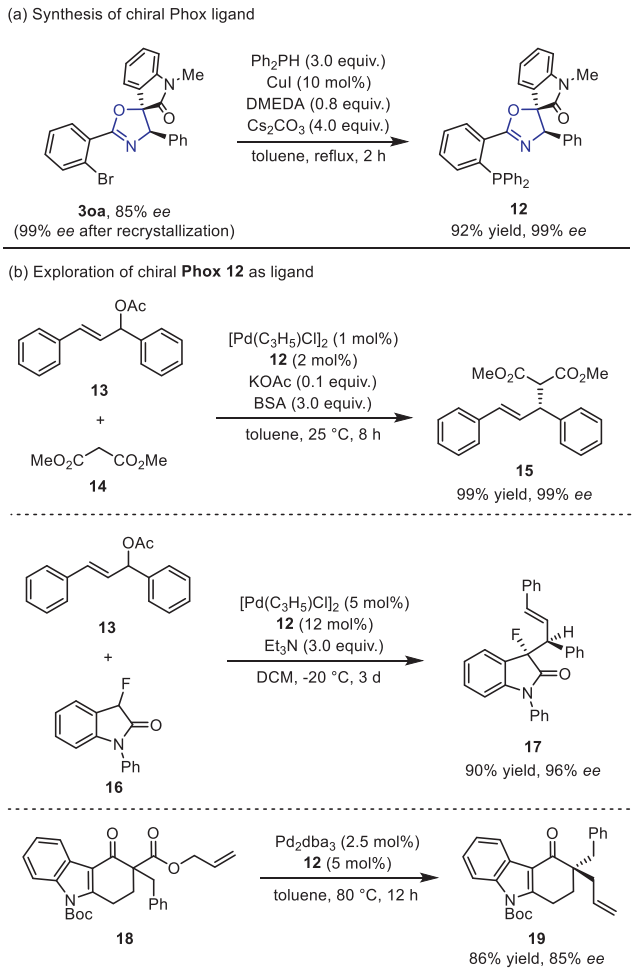
To demonstrate the potential synthetic utilities of this enantioselective [4 + 1] annulation, various transformations of the oxazoline products were explored (Scheme 4). The derivatization of the oxazoline products was first examined through Stille cross-coupling, Suzuki cross-coupling, and Buchwald-Hartwig amination reactions with allylic stannane reagent, phenylboronic acid, and benzylamine respectively. These cross-coupling reactions proceeded well and the corresponding compounds **4**–**8** were obtained in high yields with maintained enantiopurity. The conversion of the oxazoline products to 1,2-amino alcohol derivatives was conducted to further explore the synthetic potential of this reaction. Treatment of oxazoline **3aa** with 1 mol/L HCl, followed by subjecting to aqueous $NaHCO_3$ solution, delivered ring-opening 1,2-amino alcohol **9** in 82% yield without compromising enantioselectivity. Next, the transformation of oxazoline **3aa** to oxazolidine **10** was achieved smoothly without erosion of enantioselectivity through alkylation with MeOTf followed by reduction with $NaBH_4$. Oxazolidine **10** can be converted to valuable 1,2-amino alcohol **11** by treatment with PTSA.

Given the multitude of chiral oxazoline ligands and ever-increasing use of chiral phosphinooxazoline (Phox) ligands in catalytic asymmetric reactions, access to different novel Phox frameworks was of considerable interest and in great demand. To further showcase the synthetic versatility of our method, we anticipated that the enantioenriched oxazolines obtained in this study might provide a platform for the synthesis of novel types of chiral Phox ligand. Since the enantiopurity of **3oa** could be increased up to 99% *ee* after recrystallization, **3oa** could be converted to the corresponding Phox ligand **12** in 92% yield with 99% *ee via* an Ullman-type coupling (Scheme 5a). To explore the effectiveness of the obtained Phox ligand **12** in asymmetric catalysis, the preliminary application of this novel chiral Phox ligand was conducted in the allylic alkylation reaction of (*E*)−1,3-diphenylallyl acetate **13** with malonate **14**, affording **15** in 99% yield with 99% *ee* (Scheme 5b) [73,74]. We further explored the utility of chiral Phox ligand **12** in the asymmetric allylic alkylation of allyl acetate **13** with 3-fluorooxindole **16** and found that 3,3-disubstituted fluorooxindole **17** possessing vicinal chiral centers could be obtained in 90% yield with 96% *ee* [75]. Additionally, the newly designed chiral Phox ligand **12** was also successfully applied in enantioselective decarboxylative allylation of **18** to synthesize carbazolone **19**, which is a valuable chiral synthetic intermediate in alkaloid synthesis. Good enantioselectivity consistent with literature report was achieved [76]. These preliminary explorations demonstrated that the chiral Phox ligand **12** had promising potential as a new class of oxazoline ligand.

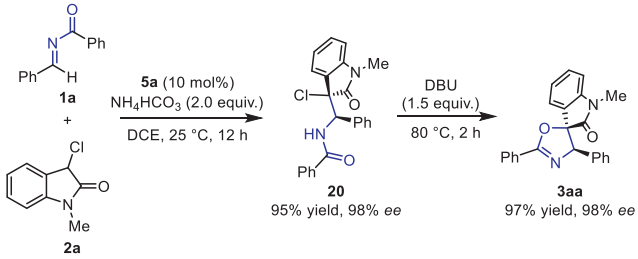
Next, to provide some experimental support for the proposed transition states, we tried to isolate the key intermediate of this reaction. The reaction of *N*-acylimine **1a** with 3-chlorooxindole **2a**



Scheme 4. Synthetic transformation of the oxazoline products.



Scheme 5. Synthesis of chiral Phox ligand and its catalytic activities.



Scheme 6. Isolation of the key intermediate.

catalyzed by chiral catalyst **5a** under ammonium bicarbonate in DCE at 25 °C was performed, and an uncyclized intermediate **20** was isolated in 95% yield with 98% *ee* (Scheme 6). Treatment of this key intermediate **20** with DBU at 80 °C could give the final cyclized oxazoline product **3aa** in 97% yield with 98% *ee*. Therefore, this reaction was a stepwise [4 + 1] annulation, and the enantiocontrol and diastereocontrol of this reaction was achieved in Mannich addition.

In conclusion, we have developed a novel organocatalytic asymmetric approach to oxazoline derivatives that proceeds through Mannich/annulation reaction of *N*-acylimines with 3-chlorooxindoles. This strategy provides an efficient and convenient method to access enantioenriched oxazolines especially valuable chiral *S,N*-oxazoline ligand as well as Ferrox ligand in high yields with excellent enantio– and diastereroselectivity. Furthermore, the reaction shows a broad substrates scope and can be applied to the expeditious assembly of valuable chiral 1,2-amino alcohols as well as chiral oxazoline-type ligand such as chiral Phox ligand owning excellent catalytic activities, demonstrating the utility of this strategy.

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

Tengfei Xuan: Methodology, Data curation. **Yuan Pan:** Methodology, Data curation. **Zhenyu Shi:** Data curation. **Yang Wang:** Writing – review & editing, Writing – original draft, Supervision, 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.cclet.2024.110352.

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