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Brønsted acid-catalyzed atroposelective desymmetrization of diamines with carboxylic acids *via* ynamide-mediated amidation

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ABSTRACT

Axially chiral diaryl ethers are unique skeletons bearing dual-axial chirality, which have broad applications in biologically active molecules. However, the catalytic atroposelective construction of diaryl ethers is very limited. Atroposelective desymmetrization of prochiral diamines is a straightforward pathway to access C–O axially chiral diaryl ethers, but the direct use of industry feedstock chemicals as desymmetrization reagents is highly challenging. Herein, we report a chiral Brønsted acid-catalyzed desymmetrizing amidation of diamines with carboxylic acids by using ynamide as coupling reagent, enabling the efficient synthesis of C–O and C–C axially chiral anilines. The synthetic utility is demonstrated by one-pot amidation with carboxylic acids, scale-up reaction, synthesis of chiral ligand and catalyst, as well as the applications in asymmetric catalysis. Importantly, this reaction represents a rare atroposelective desymmetrization of diamines with carboxylic acids.

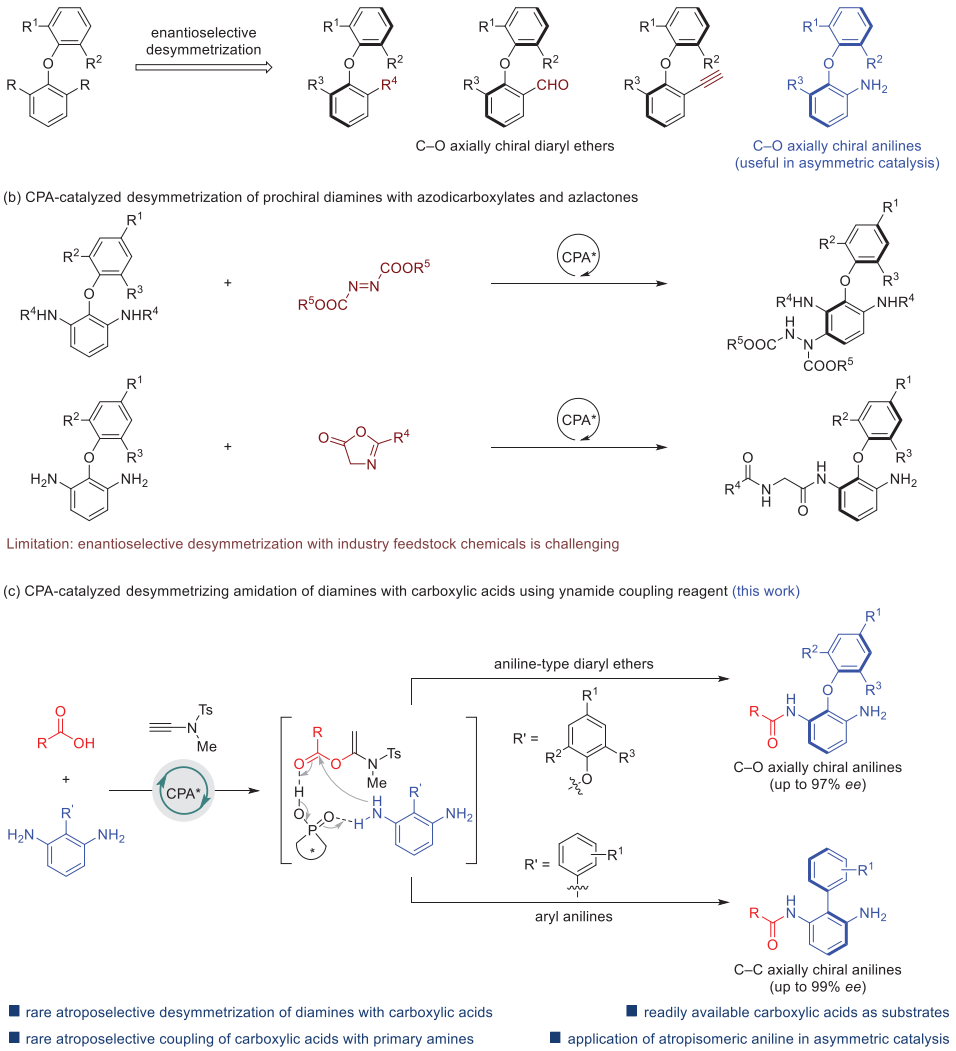
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During the past decades, axially chiral molecules have attracted considerable research interest because of their applications in asymmetric catalysis, bioactive compounds, and materials [1–5]. Axial chirality typically results from the restricted rotation about a σ bond, such as atropisomeric (hetero)biaryls, styrenes, amides, anilines, and others [6–14]. Alternatively, C–O axially chiral diaryl ethers are unique atropisomeric skeletons bearing dual-axial chirality, which have important applications in synthetic chemistry and medicinal chemistry [15–19]. Compared with the frequently reported atropisomeric biaryls, the enantioselective synthesis of axially chiral diaryl ethers is far less explored [20–22]. The more flexible dual C–O axes and relatively lower rotational barriers result in additional difficulties in the construction of axially chiral diaryl ethers [23,24]. In 2008, Clayden and co-workers disclosed a seminal work on the atroposelective synthesis of diaryl ethers by dynamic resolution with chiral sulfoxide auxiliary [25]. The more challenging catalytic enantioselective synthesis was then pioneered by Clayden's group using biocatalytic desymmetrization [26] and by Gustafson group through organocatalytic C(sp^2)–H alkylation [27], however limited scope or only moderate enantioselectivities could be obtained. Recently, various enantioselective desymmetrization reactions have been developed to achieve axially chiral diaryl ethers, such as copper(I)-catalyzed azide-alkyne cycloaddition (CuAAC) of prochiral dialkynyl diaryl ethers [28,29], cobalt-catalyzed photoreductive coupling of dialdehydes with alkynes [30], *N*-heterocyclic carbene (NHC)-catalyzed esterification of prochiral dialdehydes [31–33], chiral Brønsted acid-catalyzed transfer hydrogenation of prochiral dialdehydes [34], and chiral Brønsted acid-catalyzed Povarov reaction (Scheme 1a) [35]. In spite of these advances based on asymmetric desymmetrization, a new catalytic system that operated by metal-free conditions and simple starting materrials is still highly desirable for the enantioselective construction of C–O axially chiral diaryl ethers.

Atropisomeric amines are important chiral skeletons that have been frequently used as chiral ligands and catalysts [36,37]. Atroposelective desymmetrization of prochiral di-amines is an efficient pathway for the preparation of atropoisomeric amines [38–42]. In 2023, Yang and co-workers disclosed an elegant chiral phosphoric acid (CPA)-catalyzed electrophilic amination of prochiral diamines

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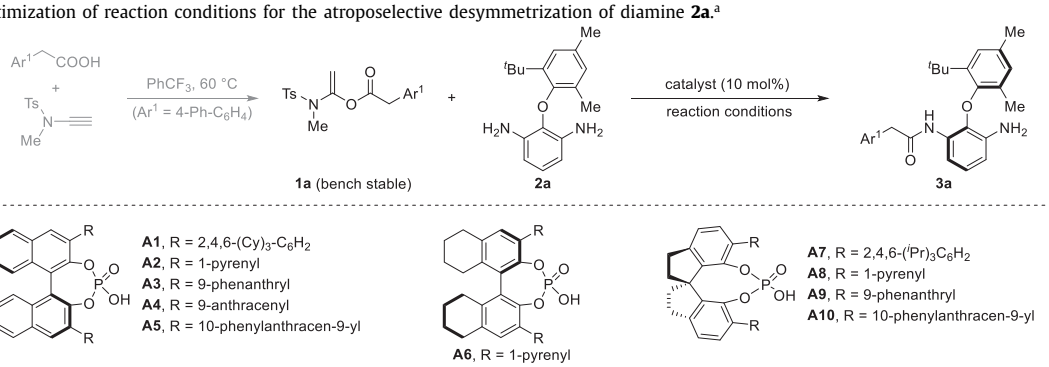
Scheme 1. Catalytic atroposelective construction of C–O axially chiral anilines.

with azodicarboxylates for the synthesis of C–O axially chiral anilines [41]. In 2024, Li and co-workers developed a CPA-catalyzed acylation of prochiral diamines with azlactones to access C–O axially chiral anilines (Scheme 1b) [42]. Despite the efficacy of these established approaches, the use of industry feedstock chemicals for the atroposelective desymmetrization of diamines is still challenging, especially for the construction of C–O axial chirality.

Carboxylic acids are important industry feedstock chemicals. The amidation of amines with carboxylic acids is a fundamental transformation in organic synthesis, and various coupling reagents have been developed to promote this dehydrative coupling process [43,44]. Due to the availability of carboxylic acids, the atroposelective amidation of amines with carboxylic acids is an ideal protocol for the synthesis of atropisomers, but it remains undeveloped for long time. By using ynamides as coupling reagents [45–48], our group recently reported the only example of CPA-catalyzed atroposelective coupling of carboxylic acids with amines, affording axially chiral amides [49]. However, the requirement of specific secondary amines and the generation of particular axially chiral amides limited the further application of this methodology. To overcome the inherent challenges in the ynamide-mediated catalytic atroposelective coupling and the synthesis of C–O axially chiral anilines, we hypothesized that ynamides could be employed as coupling reagents in the catalytic atroposelective amidation of diamines with carboxylic acids by developing new cata-lytic systems. The success of this desymmetrizing amidation relies on addressing the following key challenges: (1) The compatibility of CPA catalysts with diamine substrates is unknown, especially for primary diamines. (2) The requirement of bulky substituents for the stability of axial chirality may reduce the nucleophilicity of prochiral diamines. (3) The balance of the desymmetrization of substrates and the further kinetic resolution of products is pivotal for high yields and enantioselectivities. Therefore, efficient catalytic systems with good reactivity and enantio–control are critical for the transformation.

As part of our overarching goal to develop ynamides as versatile building blocks in organic synthesis [50–55], we have achieved a series of CPA-catalyzed enantioselective transformations of ynamides through direct activation of carbon–carbon triple bonds and resulted in diverse centrally and axially chiral heterocycles [56–61]. Inspired by these results and our recent studies on catalytic atroposelective synthesis [62–66], we envisioned to combine CPA catalysis with the atroposelective desymmetrization of diamines with carboxylic acids by using ynamides as coupling reagents, and the enantioselectivity could be controlled by multiple hydrogen bonding interactions. Here we describe the realization of such a CPA-catalyzed desymmetrizing amidation of diamines with carboxylic acids *via* ynamide mediation, leading to the

Table 1
Optimization of reaction conditions for the atroposelective desymmetrization of diamine **2a**.[a]



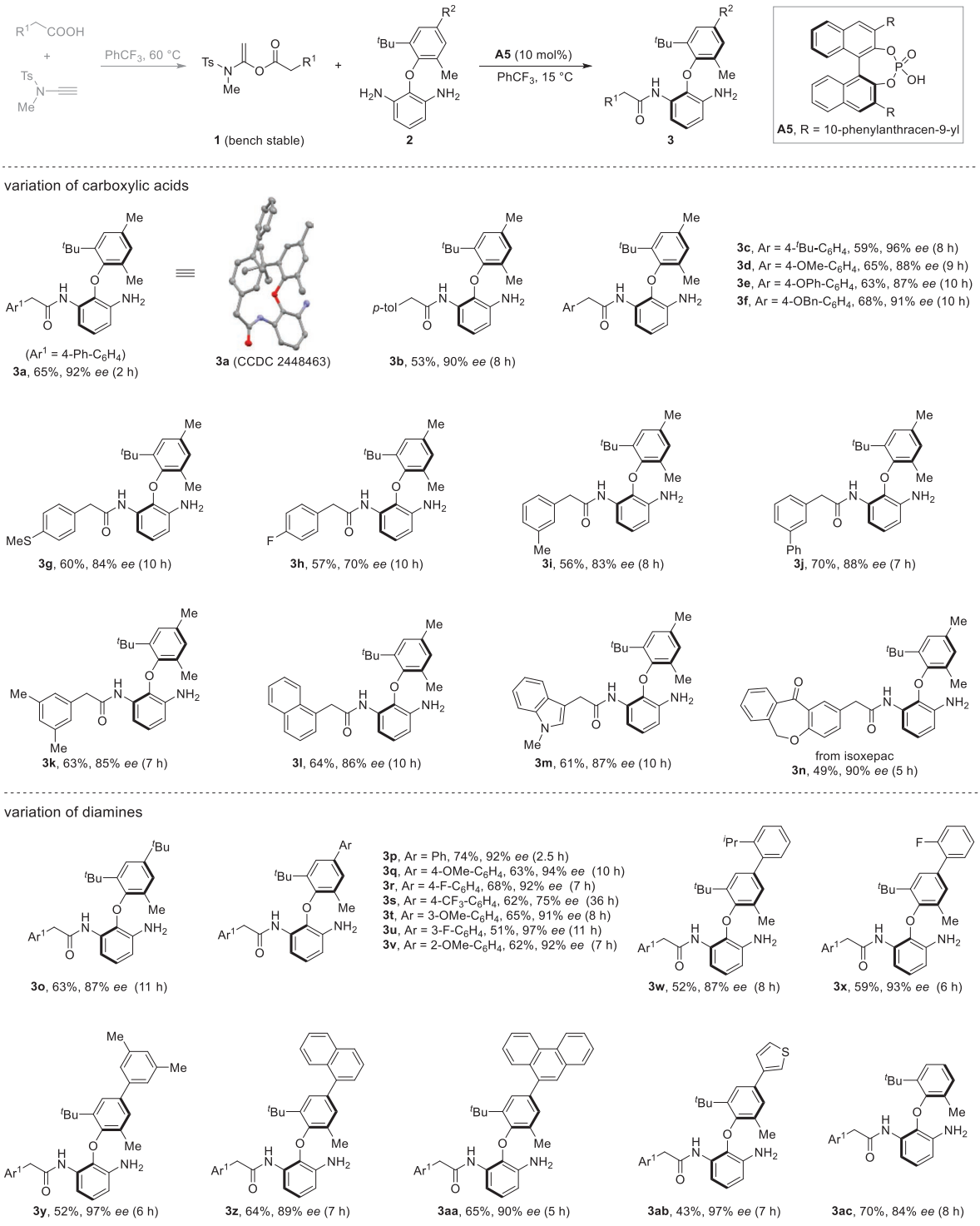
Entry	Catalyst	Solvent	Temp (°C)	Time (h)	Yield (%) [b] of **3a**	*ee* (%) [c] of **3a**
1	**A1**	CH_2Cl_2	25	4	56	20
2	**A2**	CH_2Cl_2	25	4	57	51
3	**A3**	CH_2Cl_2	25	4	63	45
4	**A4**	CH_2Cl_2	25	4	66	10
5	**A5**	CH_2Cl_2	25	4	80	75
6	**A6**	CH_2Cl_2	25	4	53	−19
7	**A7**	CH_2Cl_2	25	4	76	−47
8	**A8**	CH_2Cl_2	25	4	74	−43
9	**A9**	CH_2Cl_2	25	4	71	−39
10	**A10**	CH_2Cl_2	25	4	66	−67
11	**A5**	Et_2O	25	4	71	70
12	**A5**	THF	25	4	60	41
13	**A5**	Toluene	25	4	73	71
14	**A5**	$PhCF_3$	25	4	78	81
15	**A5**	$PhCF_3$	15	4	82	83
16[d]	**A5**	$PhCF_3$	15	4	74	87
17[e]	**A5**	$PhCF_3$	15	2	67	92

[a] Reaction conditions: **1a** (0.055 mmol), **2a** (0.05 mmol), catalyst (0.005 mmol), solvent (1.5 mL), 15–25 °C, 2–4 h, in vials.
[b] Measured by ^{1}H NMR using 1,3,5-trimethoxybenzene as the internal standard.
[c] Determined by HPLC analysis.
[d] 0.06 mmol of **1a** was used.
[e] 0.07 mmol of **1a** was used.

efficient preparation of C–O axially chiral anilines. Moreover, this atroposelective desymmetrization could also be employed in the assembly of C–C axially chiral anilines. Of note, the constructed axially chiral skeletons could be easily transformed into chiral ligands and organocatalysts, and applied in asymmetric catalysis. This reaction represents a rare atroposelective desymmetrization of diamines with carboxylic acids, as well as a rare atroposelective coupling of carboxylic acids with primary amines (Scheme 1c).

To simplify the catalytic system, this ynamide-mediated atroposelective desymmetrizing amidation of diamines with carboxylic acids was first investigated in a stepwise manner. Initially, the facile reaction of carboxylic acid with ynamide was conducted without catalyst to give a bench stable α-acyloxyenamide **1a**, which was used in the further condition optimization of atroposelective desymmetrization of diamine **2a** (Table 1). The proof-of-concept experiment was performed with a bulky BINOL-derived CPA **A1** as catalyst in CH_2Cl_2 at 25 °C, delivering the desired axially chiral diaryl ether **3a** in 56% yield with 20% *ee* (entry 1). To our delight, the exploration of different CPAs **A2**–**A5** bearing polycyclic aryl groups resulted in significantly improved enantiocontrol (entries 2–5), and 10-phenylanthracen-9-yl group-substituted CPA **A5** gave the desymmetrization product in 80% yield with 75% *ee* (entry 5). However, the switch of BINOL skeleton to H8-BINOL-derived CPA **A6** led to hugely decreased enantioselectivity (entry 6). Moreover, SPINOL-derived CPAs with 2,4,6-triisopropylphenyl **A7** and polycyclic aryl group **A8**–**A10** were also investigated, affording **3a** in 66%–76% yields with 39%–67% *ee* values (entries 7–10). Subsequent screening of different solvents, such as Et_2O, THF, toluene, and $PhCF_3$, successfully improved the enantioselectivity (entries 11–14), and $PhCF_3$ was selected as the optimal solvent (entry 14). Gratifyingly, further decreasing the reaction temperature to 15 °C and improving the equivalent of α-acyloxyenamide **1a** to 1.4 equiv. allowed the formation of axially chiral diaryl ether **3a** in 67% yield with 92% *ee* (entries 15–17).

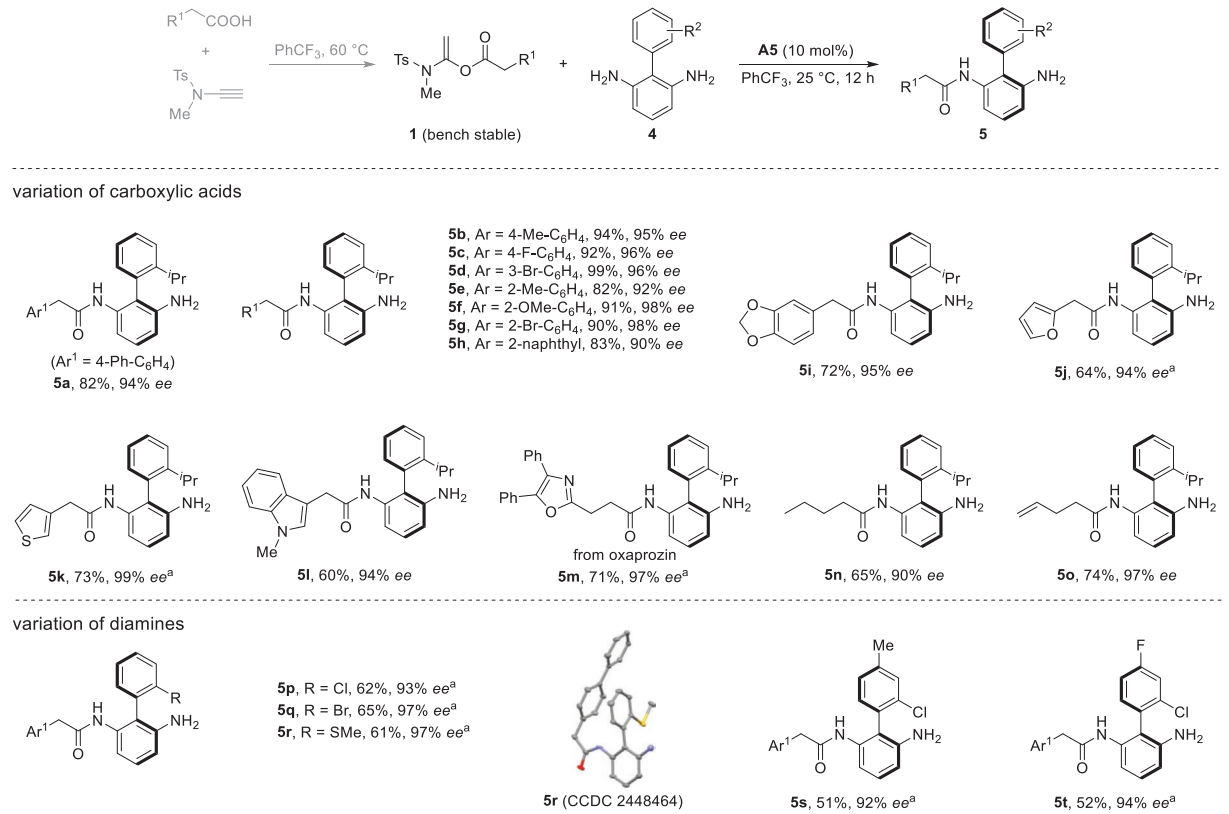
Having established the optimal reaction conditions, the substrate scope of this atroposelective desymmetrization of diamines was explored (Scheme 2). A variety of carboxylic acids were employed to react with ynamide coupling reagent to generate α-acyloxyenamides **1**, which were used in the further desymmetrizing amidation with diamines **2**. First, phenylacetic acids bearing electron-donating (Me, ^{t}Bu, OMe, OPh, OBn, and SMe) and electron-withdrawing groups (such as F and pH) on the benzene ring at the *para*- and *meta*-positions reacted smoothly to deliver the expected C–O axially chiral diaryl ethers **3a**–**3j** in 53%–70% yields with 70%–96% *ee* values. Disubstituted phenylacetic acid was found to be suitable for the transformation, furnishing the corresponding product **3k** in 63% yield with 85% *ee*. This atroposelective desymmetrization proceeded well with carboxylic acids bearing (hetero)aromatic rings, including naphthyl and Me-protected indole, affording the desired anilines in good enantioselectivities (**3l**, **3m**). Interestingly, isoxepac could be directly used as a competent carboxylic acid to give **3n** in 49% yield with 90% *ee*. Then, the scope of different diamines **2** was examined. The replacement of methyl at the *para*-position of 2-aryloxy with ^{t}Bu and phenyl groups resulted in anilines **3o** and **3p** in 63%–74% yields with 87%–92% *ee* values. Furthermore, diamines equipped with various aryl groups at the *para*-position of 2-aryloxy, including 4-OMe, 4-F, 4-CF_3, 3-OMe, 3-F, 2-OMe, 2-^{i}Pr, 2-F, and 3,5-dimethyl sub-



Scheme 2. Substrate scope for the construction of C–O axially chiral anilines **3**. Reaction conditions: **1** (0.14 mmol), **2** (0.1 mmol), **A5** (0.01 mmol), $PhCF_3$ (3.0 mL), 15 °C, 2–36 h, in vials; yields were those for the isolated products; ee values were determined by HPLC analysis.

stituted phenyl groups, as well as 1-naphthyl, 9-phenanthryl and 3-thienyl groups, reacted efficiently to produce the corresponding **3q**–**3z**, **3aa** and **3ab** in 43%–68% yields with 75%–97% *ee* values. In addition, diamine substrate bearing disubstituted phenyl group was evaluated in this reaction, and the expected product **3ac** was generated in 70% yield with 84% *ee*. The absolute configuration of **3a** was confirmed by X-ray diffraction. Details can be found in Supporting information

To further explore the capacity of this atroposelective desymmetrization, we proposed to further extend this methodology to the synthesis of C–C axially chiral anilines [38–40]. After systematic evaluation of reaction conditions (see Supporting information for details), the ynamide-mediated amidation of diaryl anilines with carboxylic acids was first conducted in a stepwise process. As shown in Scheme 3, a variety of phenylacetic acids with both electron-donating and electron-withdrawing substituents at differ-



Scheme 3. Substrate scope for the construction of C–C axially chiral anilines **5**. Reaction conditions: **1** (0.11 mmol), **4** (0.1 mmol), **A5** (0.01 mmol), $PhCF_3$ (3.0 mL), 25 °C, 12 h, in vials; yields were those for the isolated products; *ee* values were determined by HPLC analysis. [a]**1** (0.14 mmol).

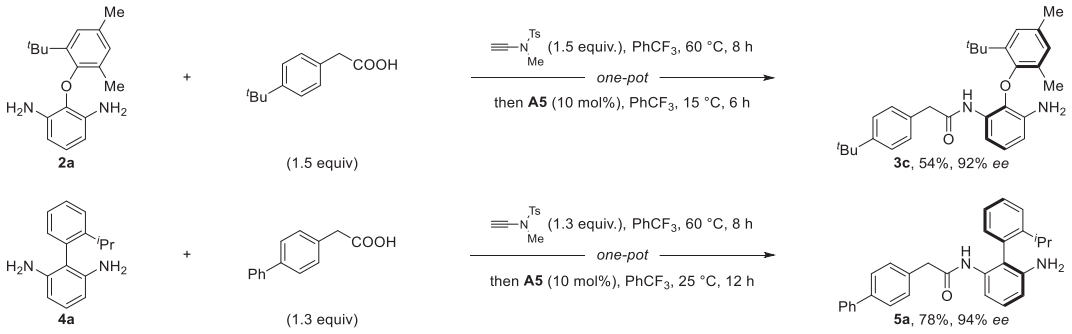
ent positions of phenyl ring, such as Me, OMe, pH, F, and Br, could undergo this atroposelective amidation efficiently to generate atropisomeric anilines **5a**–**5g** in 82%–99% yields with 92%–98% *ee* values. Other (hetero)aromatic moieties, including 2-naphthyl (**5h**), 1,3-benzodioxole (**5i**), furan (**5j**), thiophene (**5k**), and Me-protected indole (**5l**) were all compatible in the transformation, delivering the desired products in 60%–83% yields with 90%–99% *ee* values. Oxaprozin could also be transformed into the corresponding axially chiral product **5m** smoothly. The arylacetic acid could be replaced with valeric acid and 4-pentenoic acid to give **5n** and **5o** in 65%–74% yields with 90%–97% *ee* values. Next, we turned to explore the scope of diamines **4**. The replacement of isopropyl on diaryl aniline substrate with Cl, Br, and SMe, successfully produced the target **5p**–**5r** in 61%–65% yields with 93%–97% *ee* values. Moreover, diamines equipped with disubstituted aryl group were also suitable substrates for the amidation reaction, affording **5s** and **5t** in moderate yields with high enantioselectivities. The absolute configuration of **5r** was confirmed by single crystal X-ray crystallographic analysis (see Supporting information for details). This protocol provides a concise way for the construction of axial chirality [67–73].

Encouraged by the stepwise reactions, the one-pot desymmetrizing amidation of diamines with carboxylic acids was successfully realized by ynamide coupling reagent. As shown in Scheme 4, the one-pot atroposelective amidation of diamine **2a** with 2-(4-(*tert*–butyl)phenyl)acetic acid proceeded smoothly to deliver C–O axially chiral aniline **3c** in 54% yield with 92% *ee*, and the one-pot reaction of diamine **4a** with 2-([1,1′-biphenyl]−4-yl)acetic acid afforded C–C axially chiral aniline **5a** in 78% yield with 94% *ee*. Therefore, this CPA-catalyzed strategy enables the effective atroposelective amidation of diamines with readily available carboxylic acids.

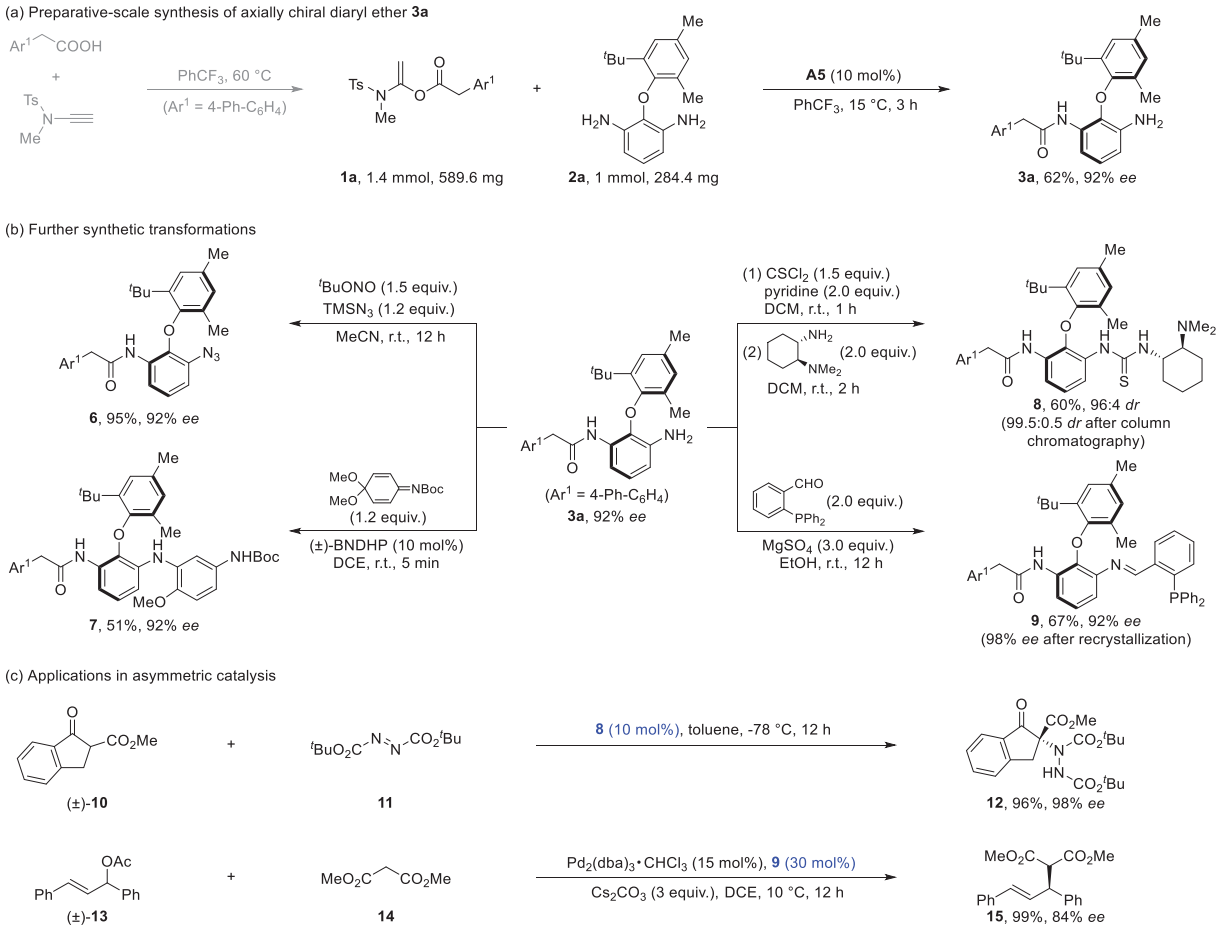
To explore the synthetic utility of this strategy, the scale-up reaction of α-acyloxyenamide **1a** with diamine **2a** was performed, and the desired C–O axially chiral aniline **3a** could be obtained in 62% yield with 92% *ee* (Scheme 5a). The aniline moiety enabled diverse transformations, such as diazotisation reaction and Michael addition with 1,1′-binaphthyl-2,2′-diyl hydrogenphosphate (BNDHP) to yield axially chiral diaryl ethers **6** and **7** efficiently. The treatment of free aniline with thiophosgene ($CSCl_2$) and (1*S*,2*S*)-*N*,*N*′-dimethylcyclohexane-1,2-diamine delivered a diastereomeric tertiary amine-thiourea bifunctional catalyst **8**. Furthermore, axially chiral free amine **3a** could undergo condensation with 2-(diphenylphosphino)benzaldehyde to synthesize the C–O axially chiral *P*,*N*-ligand **9**. Notably, these transformations proceeded without erosion of enantiopurity (Scheme 5b).

The application of above C–O axially chiral skeletons in asymmetric catalysis was then investigated (Scheme 5c). Importantly, thiourea **8** could be used as a competent organocatalyst for the enantioselective Michael addition of β-ketoester **10** with di–*tert*–butyl azodicarboxylate (DBAD) **11**, affording the α-hydrazination product **12** in 96% yield with 98% *ee*. Moreover, axially chiral phosphine **9** was demonstrated to be an effective ligand in palladium-catalyzed asymmetric Tsuji-Trost allylation of 1,3-diphenylallyl acetate **13** and dimethyl malonate **14**, and the desired product **15** could be isolated in almost quantative yield with 84% *ee*. These results indicate the potential application of the constructed axially chiral aniline skeletons in the field of asymmetric catalysis.

To probe the reaction mechanism and origin of enantioselectivity, a series of control experiments were carried out. As shown in Scheme 6a, the desymmetrization of diamine **2a** with 0.55 equiv. of α-acyloxyenamide **1a** was conducted, leading to C–O axially chiral aniline (*R*)-**3a** in 39% yield with 73% *ee*, accompanied with trace amount of diamidation product **16** ($K_R/K_S = 6.4{:}1$). The kinetic res-



Scheme 4. One-pot atroposelective desymmetrizing amidation of diamines with carboxylic acids.

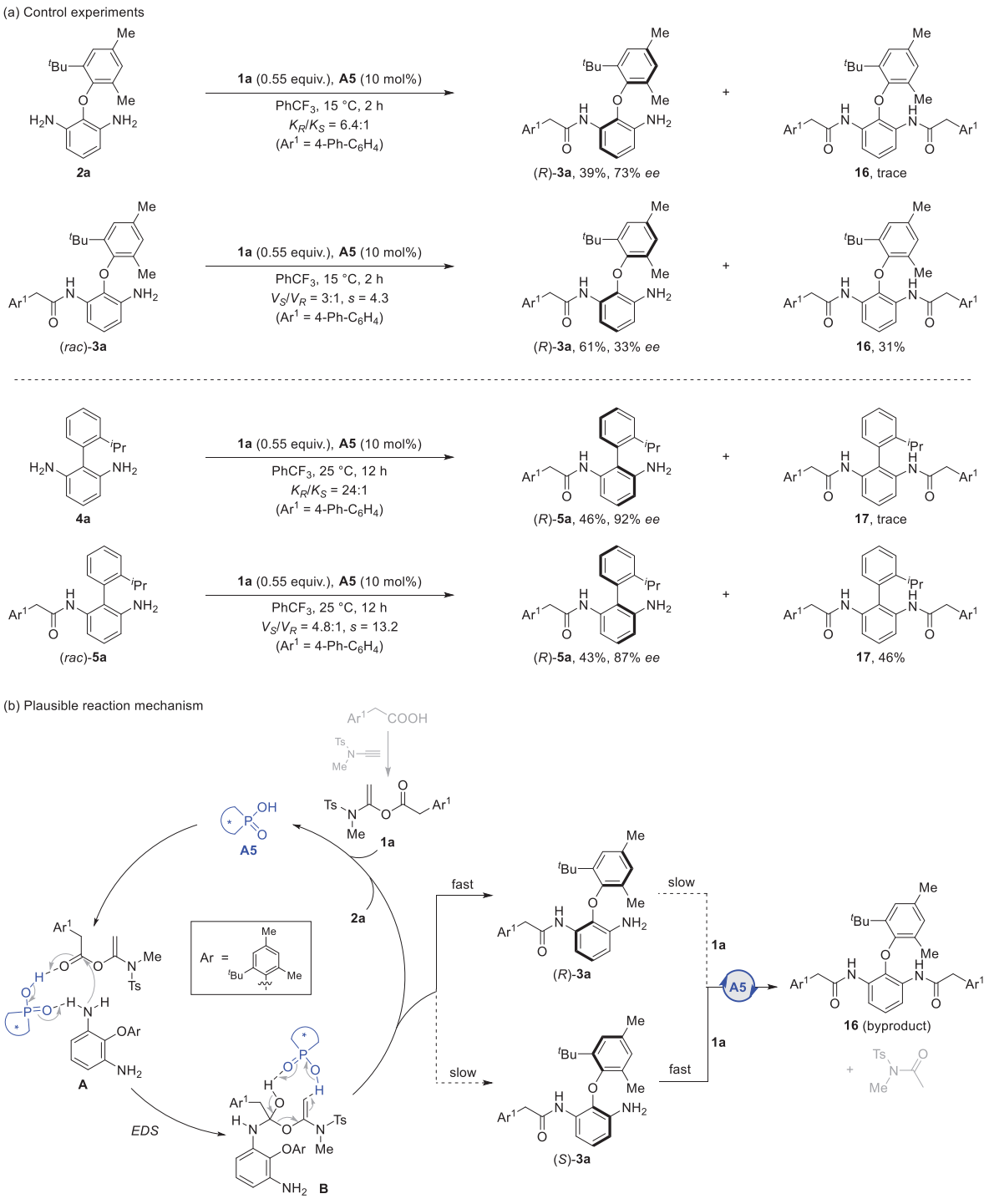


Scheme 5. Transformations and applications of C–O axially chiral anilines.

olution of racemic **3a** with 0.55 equiv. of α-acyloxyenamide **1a** afforded (*R*)-**3a** in 61 % yield with 33 % *ee*, as well as diamidation product **16** in 31 % yield ($V_S/V_R = 3{:}1$, the selective factor $s = 4.3$). These results demonstrate that desymmetrization is the major contributor for enantiocontrol, and the following kinetic resolution improved the enantioselectivity of **3a**. Similarly, the desymmetrization of diamine **4a** and kinetic resolution of racemic **5a** were also performed, indicating that desymmetrization is the key contributor for enantiocontrol, and the following kinetic resolution slightly improved the enantioselectivity of **5a** ($K_R/K_S = 24{:}1$; $V_S/V_R = 4.8{:}1$, the selective factor $s = 13.2$).

Based on the control experiments and the previous studies on ynamide-mediated coupling reaction [49], the plausible reaction mechanism is postulated (Scheme 6b). After the synthesis of α-acyloxyenamide **1a** from the reaction of carboxylic acid with ynamide, CPA catalyst **A5** promotes the atroposelective nucleophilic addition of diamine **2a** onto α-acyloxyenamide **1a** *via* double hydrogen bonding interactions (intermediate **A**) to form tertiary alcohol-type intermediate **B**, which is the enantio–determining step (EDS). Subsequently, a hydrogen bonding-assisted proton transfer takes place to afford axially chiral diaryl ether (*R*)-**3a** and regenerate the catalyst. The enantioselectivity of (*R*)-**3a** is further improved by the following kinetic resolution of undesired (*S*)-**3a** through the second amidation, which generates diamidation byproduct **16** and sulfonyl-protected *N*-methylacetamide. Of note, the sulfonyl-protected *N*-methylacetamide byproduct can be easily converted into ynamide coupling reagent and applied into other reactions [50].

In conclusion, a Brønsted acid-catalyzed atroposelective desymmetrizing amidation of diamines with carboxylic acids has been



Scheme 6. One-pot atroposelective desymmetrizing amidation of diamines with carboxylic acids.

developed by using ynamide as coupling reagent, leading to the assembly of C–O and C–C axially chiral anilines in high enantioselectivities. Importantly, the one-pot atroposelective amidation of diamines with carboxylic acids was successfully realized *via* ynamide-mediation, which showed good tolerance towards primary diamines. This reaction represents a rare atroposelective desymmetrization of diamines with carboxylic acids. Mechanistic studies indicate that this atroposelective amidation involves a desymmetrization/kinetic resolution cascade sequence. The scale-up reaction and synthetic transformations led to the constructions of C–O axially chiral organocatalyst and *P,N*-ligand, which could be applied in asymmetric catalysis. Further investigation into the ynamide-mediated enantioselective coupling strategy for the efficient synthesis of chiral functional molecules is underway in our laboratory.

CRediT authorship contribution statement

Guang-Hui Wang: Data curation. **Jia-Tian Jiang:** Data curation. **Jian Yang:** Data curation. **Ze-Shu Wang:** Data curation. **Gongde Wu:** Formal analysis. **Long-Wu Ye:** Supervision. **Bo Zhou:** Writing

– review & editing, Supervision, Project administration, Funding acquisition, Conceptualization.

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.

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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.2025.112278.

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