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Dynamic kinetic stereodivergent transformations of propargylic ammonium salts *via* dual nickel and copper catalysis

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ABSTRACT

The dynamic kinetic asymmetric transformation of racemic propargylic ammonium salts with prochiral aldimine esters through a stereodivergent propargylation is catalyzed by dual nickel and copper catalysis. Thus, a diverse range of optically active α-quaternary amino esters were produced *via* C–N bond cleavage with high reaction efficiency and stereoselectivity (up to >99% *ee*). By selection of the appropriate pairwise combination of catalyst configurational isomers, all four possible stereoisomers of the corresponding propargylation products are obtained in high yields with excellent regio-, diastereo-, and enantioselectivities.

Stereocontrolled carbon-carbon bond forming reactions at the propargylic position are particularly desirable, and the resulting products can be easily subjected to further modifications with potential applications in materials, organic chemistry and pharmaceuticals [1–4]. Recently, considerable efforts have been invested in the development of distinct transition metal-mediated species that enable asymmetric transformations of propargylic substrates bearing an internal alkyne group in a regiocontrolled, stereocontrolled and diversity-oriented manner [5–25]. Efficient selective cleavage of the C–N bonds [26–28] and further synthetic applications in transition metal-catalyzed propargylation reaction would be very attractive due to the potential opportunity of developing novel methodologies in synthesis and chemical processes. Remarkably, the Tortosa group made a seminal contribution to the development of a regio- and stereospecific copper-catalyzed substitution reaction of optical active propargylic ammonium salts (Scheme 1a) [29,30]. Recently, the Oestreich group have developed elegant copper-catalyzed nucleophilic silylation of organoammonium salts to obtain trisubstituted allenes (Scheme 1b) [31]. Although significant progress has been made, examples of transition metal-catalyzed dynamic kinetic asymmetric transformation (DyKAT) [32,33] of propargylic ammonium salts are still rare, especially in stereo-divergent manner. In the context, developing simple and direct methodology for achieving the catalytic asymmetric propargylic substitution reaction of racemic propargylic ammonium salts is highly desirable. We anticipated that nickel catalysts would enable the cross coupling of a wide variety of propargylic ammonium salts *via* cleavage of C–N bonds [34–45]. Herein, we report a Ni/(*R*)-Binap catalyst system for the DyKAT of racemic propargylic ammonia salts.

Nowadays, an increasing amount of attention is being focused on the construction of complete stereoisomers of a chiral molecule containing multiple contiguous stereocenters with full control of absolute and relative stereochemical configuration, and the use of two chiral catalysts to activate synergistically two substrates and dictate the configuration of the stereocenters has emerged as a powerful strategy in chemical synthesis [46–50]. Recently, the bimetallic catalytic system [51–56], which can set the chiral elements simultaneously at both the electrophile and nucleophile, offers a unique opportunity for exploration of stereodivergent transformations [57–67]. Typically, chiral copper complexes assisted the conversion of aldimine esters **2** to the nucleophilic *N*-metalated azomethine ylides [68] that have a well-defined geometry and can react with high facial selectivity in the stereodivergent alkylation reactions disclosed by the groups of Zhang [58,65,66], Wang [59,63,64,67] and Zi [61]. We reasoned that the asymmetric dual bimetallic system *via* the combination of nickel and copper catalysis can be extended to realize the stereo-divergent propargylic alkylation reactions of racemic propargylic ammonium salts and aldimine esters (Scheme 1c) [69]. Here we report the Ni/Cu dual

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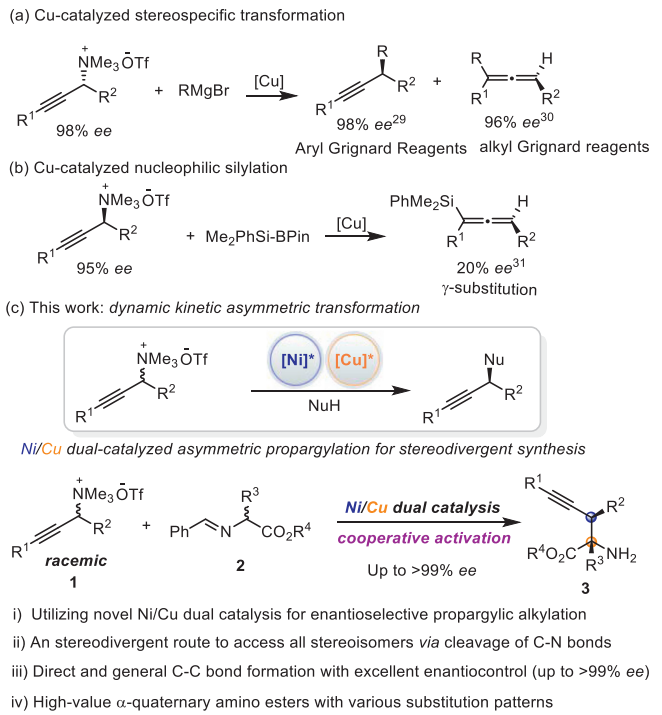
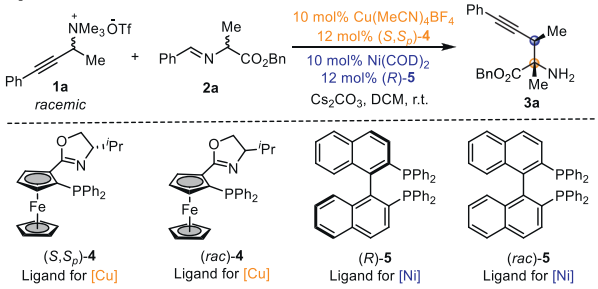


Scheme 1. Strategies for dual Ni/Cu-catalyzed dynamic kinetic stereodivergent transformations of propargylic ammonium salts.

catalytic approach for the expeditious construction of α-quaternary amino esters [70–72] in a DyKAT process with excellent levels of regio-, diastereo-, and enantioselectivity.

To substantiate the aforementioned reaction design, we began our investigation into the Ni/Cu-catalyzed asymmetric alkylation of racemic propargylic ammonium salt **1a** with aldimine ester **2a** (Table 1). The combined use of a chiral copper complex modified

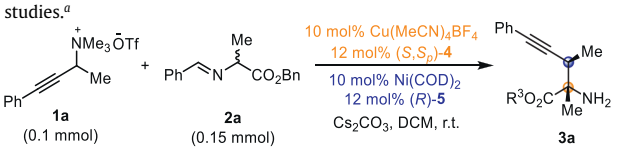
Table 1
Optimization of the reaction conditions.[a]



Entry	Variation from the standard conditions	Yield (%)[b]	*dr*[c]	*ee* (%)[d]
1	None	85	18:1	>99
2	Without $Cu(MeCN)_4BF_4$	39	1:1	89
3[e]	Without ligand (S,S_p)-**4**	45	6:1	97
4	Without $Ni(COD)_2$	nr	–	–
5	Without ligand (*R*)-**5**	nr	–	–
6	Without Cs_2CO_3	nr	–	–
7	*rac*-**4** instead of (S,S_p)-**4**	85	2:1	64
8	*rac*-**5** instead of (*R*)-**5**	81	2:1	50

[a] Reactions were performed by using $Cu(MeCN)_4BF_4$ (10 mol%), (S,S_p)-**4** (12 mol%), $Ni(COD)_2$ (10 mol%), (*R*)-**5** (12 mol%), **1a** (0.24 mmol, 2.4 equiv.), **2a** (0.1 mmol, 1.0 equiv.), and Cs_2CO_3 (0.3 mmol) in dichloromethane (DCM) at r.t.; hydrolysis with HCl (1 mol/L, 4 mL).
[b] Isolated yields after column chromatography are shown.
[c] Determined by ^1H NMR spectroscopy.
[d] Determined by HPLC analysis.
[e] In the presence of (*R*)-**5** (10 mol%).

Table 2
Investigation of chiral propargylic ammonium salts for preliminary mechanistic studies.[a]



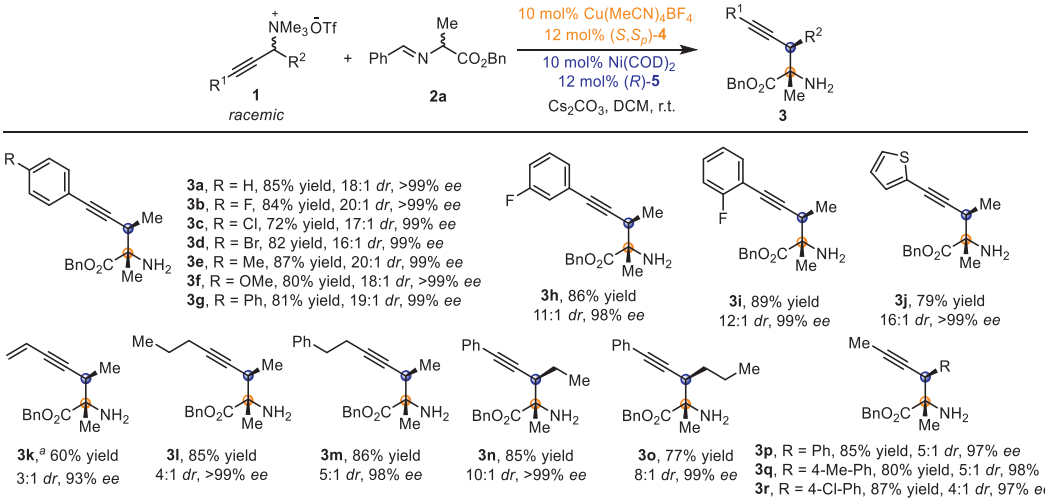
Entry	**1**	Yield (%)[b]	*dr*[c]	*ee* (%)[d]
1	*rac*-**1a**	78	>20:1	>99
2	(*S*)-**1a**	70	>20:1	>99
3	(*R*)-**1a**	74	>20:1	>99
4[e]	(*R*)-**1a**	45	1:1	0

[a] Reactions were performed by using $Cu(MeCN)_4BF_4$ (10 mol%), (S,S_p)-**4** (12 mol%), $Ni(COD)_2$ (10 mol%), (*R*)-**5** (12 mol%), **1a** (0.1 mmol, 1.0 equiv.), **2a** (0.15 mmol, 1.5 equiv.), and Cs_2CO_3 (0.1 mmol) in dichloromethane (DCM) at rt; hydrolysis with HCl (1 mol/L, 4 mL).
[b] Isolated yields after column chromatography are shown.
[c] Determined by ^1H NMR spectroscopy.
[d] Determined by HPLC analysis.
[e] The reaction was conducted by using $Ni(COD)_2$ (10 mol%), (*rac*)-**5** (12 mol%), (*R*)-**1a** (0.24 mmol, 2.4 equiv.), **2a** (0.1 mmol, 1.0 equiv.), and Cs_2CO_3 (0.3 mmol) in DCM at r.t.

with the Phosferrox ligand (S,S_p)-**4** and a chiral nickel complex derived from bidentate phosphine ligand (*R*)-**5** successfully afforded the desired product **3a** in high yield with excellent stereocontrol at room temperature (entry 1, 85% yield, 18:1 *dr*, >99% *ee*). With the aim of gaining more insights into the synergistic effect of the bimetallic catalysis, a series of control experiments were carried out (entries 2–8). In the absence of Cu catalyst or ligand (S,S_p)-**4**, the efficiency and the selectivities of the reaction deceased dramatically (entries 2 and 3). Notably, no reaction was observed in the absence of other reaction components (the nickel catalyst, ligand (*R*)-**5**, or Cs_2CO_3) (entries 4–6). With the use of racemic ligands **4** and **5**, respectively, large variations in the diastereo- and enantioselectivity were observed (entries 7 and 8).

To investigate the reaction mechanism, enantiopure propargylic ammonium salts (*R*)-**1a** and (*S*)-**1a** were prepared and subjected to the catalysis conditions, respectively (Table 2). Notably, the propargylation with the enantioenriched ammonium salts **1a** led to the product **3a** with apparently the same results as when racemic salts *rac*-**1a** was applied (entries 1–3). Furthermore, Ni/*rac*-**5** catalyst can individually catalyze the propargylation reaction of (*R*)-**1a** with **2a** leading to the racemic product **3a** albeit with low yield (entry 4). Consequently, the combination of copper/(S,S_p)-**4** complex and nickel/(*R*)-**5** complex was essential for this DyKAT process, which verifies the superiority of synergistic catalysis in the propargylation alkylation reactions.

We then investigated the substrate range of propargylic ammonium salts under the optimum conditions (Scheme 2). A broad variety of racemic propargylic ammonium salts, including those with electron-withdrawing or donating substituents on the benzene ring, underwent the dual catalytic transformation with favorable yields and outstanding diastereo- and enantioselectivities (**3b**-**3i**). Encouragingly, the enantioselective propargylic alkylation could be successfully conducted on a large scale, consistently yielding **3a** with comparable yields, diastereo-, and enantioselectivities (0.70 g, 70% yield, 9:1 *dr*, 98% *ee*). The reaction also tolerated propargylic ammonium salts bearing heteroaromatic substituents, as evidenced by the production of the thiophenesubstituted product **3j** with 79% yield and high stereoselectivity (16:1 *dr*, >99% *ee*). Furthermore, propargylic ammonium salts containing an alkenyl group were compatible with this approach, producing the required compounds in moderate yields and with high enantioselectivity using THF as solvent (**3k**). Notably, dialkyl-substituted propargylic carbonates did not compromise reaction efficiency or enantiocontrol

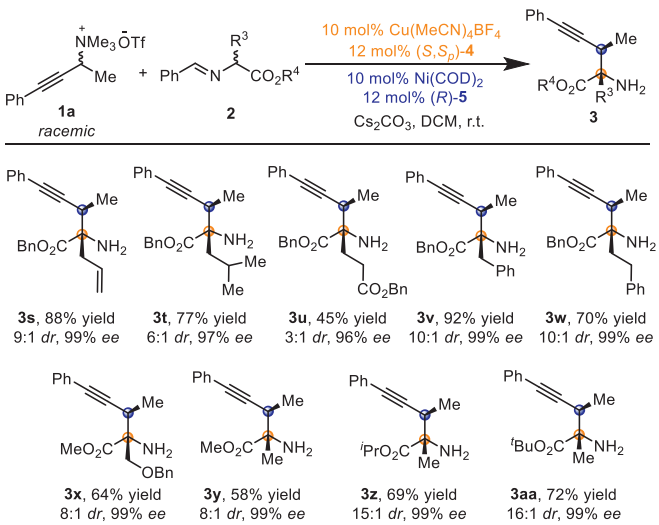


Scheme 2. Substrate scope of propargylic ammonium salts. Reactions were performed by using $Cu(MeCN)_4BF_4$ (10 mol%), (S,S_p)–**4** (12 mol%), $Ni(COD)_2$ (10 mol%), (R)–**5** (12 mol%), **1** (0.24 mmol, 2.4 equiv.), **2a** (0.1 mmol, 1.0 equiv.), and Cs_2CO_3 (0.3 mmol) in dichloromethane (DCM) at r.t.; hydrolysis with HCl (1 mol/L, 4 mL). [a] THF instead of DCM.

(**3l** and **3m**). Propargylic carbonates bearing various substituents on the propargylic carbon atom performed efficiently in the asymmetric alkylation reaction (**3n**-**3r**). These findings highlight the versatility and potential of this method in the synthesis of diverse chiral propargylic compounds.

In addition, we examined the versatility of this remarkable reaction by investigating the impact of various substituents on aldimine esters **2** (Scheme 3). Remarkably, a diverse range of aldimine esters **2** served as excellent nucleophiles in the propargylic alkylation process, delivering the desired products with high yields and stereoselectivities (**3s**-**3x**). Furthermore, variations in the ester group of **2** had no apparent impact on the reaction outcomes (**3y**-**3aa**), further demonstrating the robustness and flexibility of the methodology.

The propargylation reactions of racemic propargylic ammonium salts to access all possible stereoisomers would be challenge (Scheme 4). Only low diastereoselectivity of the desired product

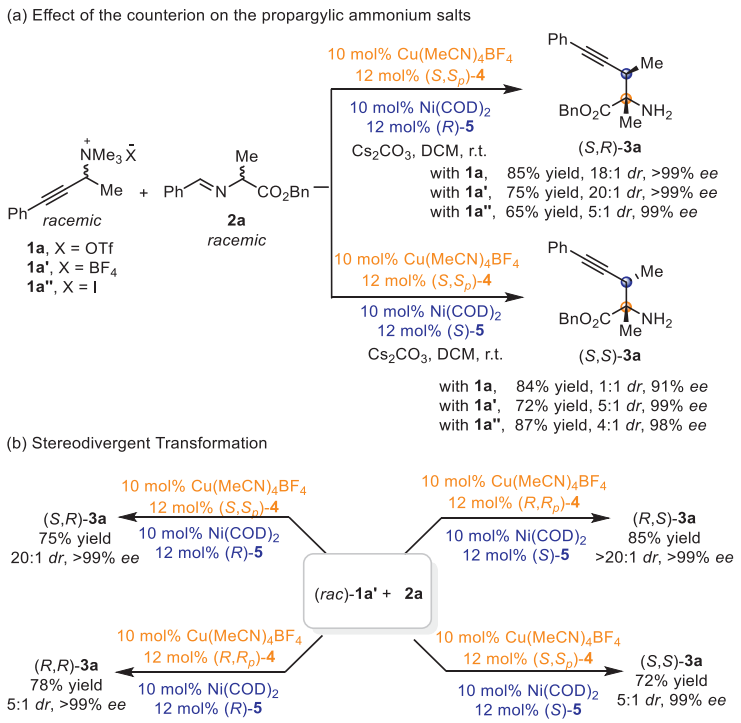


Scheme 3. Substrate scope of aldimine esters. Reactions were performed by using $Cu(MeCN)_4BF_4$ (10 mol%), (S,S_p)–**4** (12 mol%), $Ni(COD)_2$ (10 mol%), (R)–**5** (12 mol%), **1a** (0.24 mmol, 2.4 equiv.), **2** (0.1 mmol, 1.0 equiv.), and Cs_2CO_3 (0.3 mmol) in dichloromethane (DCM) at r.t.; hydrolysis with HCl (1 mol/L, 4 mL).

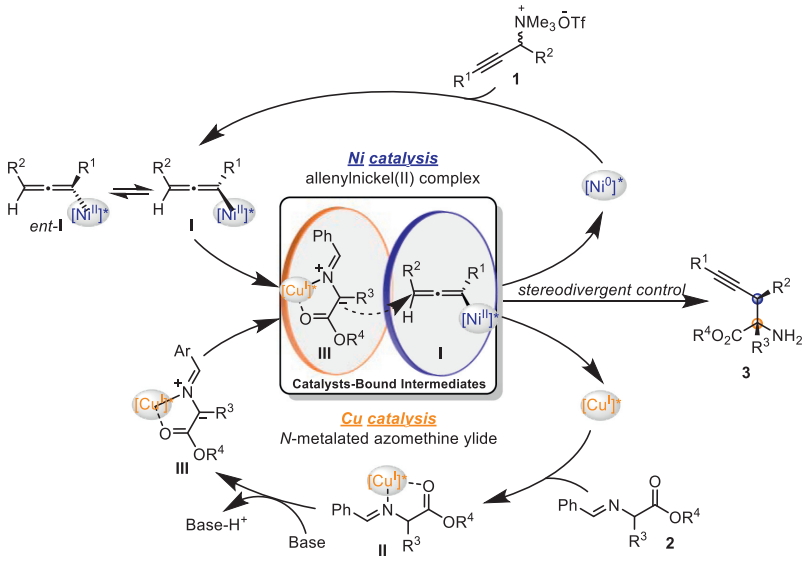
(*S*,*S*)-**3a** was observed using triflate counterion **1a** substrate when the reaction was conducted with (*S*)-**4** as the ligand for nickel and (S,S_p)-**5** as the ligand for copper for the bimetallic system (84% yield, 1:1 *dr*, 91% *ee*). Subsequently, the influence of the counterion on the propargylic ammonium salts was examined, and **1a'** with tetrafluoroborate anion was proved to be efficient substrate in turning the diastereoselectivity (72% yield, 5:1 *dr*, >99% *ee*). To further demonstrate the stereodivergence of this Ni/Cu dual-catalyzed alkylation, the enantioselective propargylation of **1a'** with aldimine ester **2** was conducted with four different pairs of enantiomers of the nickel catalyst and the copper catalyst under otherwise identical conditions (Scheme 4). All four stereoisomers of the desired products **3a** were produced in good yields, with high diastereo- and enantioselectivity. These results strongly indicate that each chiral metal complex is independently responsible for a different stereogenic center in the cooperative propargylation reactions.

A plausible mechanistic cycle, in which nickel catalysis intertwines with copper catalysis for the DyKAT of racemic propargylic ammonium salts with prochiral aldimine esters, is outlined in Scheme 5. In this Ni/Cu dual catalytic system, chiral nickel complexes combine transiently with racemic propargylic ammonium salts **1** cleaving the C–N bonds to generate the electrophilic allenylnickel(II) intermediates **I** [26-28,73-77]. Subsequent nucleophilic addition of the Cu-coordinated azomethine ylides **III** [78] onto the allenylnickel(II) intermediates **I** [79–81] *via* a catalytic enantio–convergent pathway would provide a novel stereoselective route to access chiral α-tertiary amines through propargylation process. Importantly, the configuration of the adjacent stereocenters could be independently dictated with these two independent catalytic systems (Scheme 5).

In conclusion, our strategy represents a chiral Ni/Cu dual catalysis for the DyKAT of racemic propargylic ammonium salts with prochiral aldimine esters. Taking advantage of the synergistic effects for enhanced catalytic properties, we designed the stereodivergent propargylic alkylation to afford the synthetic useful α-quaternary amino esters with broad scope, outstanding efficiency, uniformly excellent yield, high diastereoselectivity, and excellent enantioselectivity. Furthermore, the novel stereodivergent Ni/Cu dual catalysis can provide a unified route, applying racemic internal propargylic carbonates to access all four stereoisomers of corresponding products. Remarkably, the individual activation role of each chiral metal complex has the potential to be a powerful plat-



Scheme 4. Stereodivergent transformation and synthetic versatility of the dual catalytic system.



Scheme 5. Proposed mechanism.

form for the development of a wide range of broadly useful stereocontrolled reactions.

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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