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Regio- and enantioselective conjugate addition of β-nitro α,β-unsaturated carbonyls to construct 3-alkenyl disubstituted oxindoles

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ABSTRACT

Reversal of regioselectivity in the catalytic asymmetric conjugate additions of 3-substituted oxindoles to β-nitroenones or β-nitroacrylates was established with chiral scandium catalysts. It enabled the construction of functionalized 3,3-disubstituted oxindoles, including terminal and internal vinyl groups in excellent yields and *ee* values.

The asymmetric catalytic conjugate addition to electron-deficient alkenes is one of the most useful synthetic routes for the construction of optically active compounds [1–11]. Among the various Michael acceptors, the nitroalkenes have drawn more interests not only due to the transformation of nitro group into other functional groups [12–23], but also formation of formal alkenylation derivative *via* the elimination under suitable reaction conditions [24–27]. In recent years, the asymmetric catalytic conjugate of rich functionalized β-nitro α,β-unsaturated carbonyls have been studied (Scheme 1a), which has higher reactivity as pull-pull olefins due to the global electron depletion [28,29]. Nevertheless, the doubly activated olefins with different vicinal electron-withdrawing groups might raise the regioselectivity. Hu's group reported chiral Rh^{I}-diene catalyzed asymmetric three-component reaction of aryldiazoacetates, aromatic amines and β-nitroacrylates to obtain γ-nitro-α-amino succinates [30]. Asymmetric conjugate addition of metal alkyl reagents or arylboronic acids to β-nitroacrylates to generate β-amino acid precursors was also realized by several research groups [31–34]. On the other hand, formal α-alkenylations of aldehydes or β-ketoesters have been reported by Jørgensen [35] and Mukherjee [36], respectively, using β-nitroacrylates (Scheme 1b, i) and β-nitroenones (Scheme 1b, ii). However, in those cases, α-selective conjugate addition occurred which might provide a bias that this is the dominated regioselectivity in nucleophilic addition of pull-pull olefines.

Oxindoles represent a privileged backbone existing in many natural products and bioactive compounds, and also important building blocks in organic synthesis [37–43]. The nucleophilic conjugate addition of 3-substituted oxindoles increased the diversity of oxindoles, and the use of but–2-ynedioate [44,45], alkynones [46], or β-haloloalkenes [47] as the Michael acceptors could give vinylic substituted oxindoles. Nevertheless, asymmetric alkenylation to construct quaternary oxindoles with an *exo*-methylene-substitution is elusive. In view of the biological properties of oxindoles, the interesting regio-selectivity of nitroolefins, as well as the elimination tendency to generate formal alkenylated product, we carried out regio-and enantioselective conjugate addition of β-nitro α,β-unsaturated carbonyls with 3-substituted oxindoles. Here, we found that in the presence of chiral *N,N′*-dioxide/scandium complexes [48–52], the reaction with β-nitroenones occurred *via* a β-selective addition, in which benzoyl group acts as a stronger directing group (Scheme 1c, i); whereas the reaction with β-nitroacrylates occurred *via* a α-selective addition where nitro-group acts as the activated group (Scheme 1c, ii). Both terminal and internal vinyl substituted oxindoles could be obtained in excellent yield and enantioselectivity in the presence of suitable bases.

In the initial study, we chose phenyl β-nitroenone **B1** as the Michael acceptor and 3-benzylindolinone **A1** as the nucleophile to construct quaternary carbon center (Table 1). Firstly, metal salts co-

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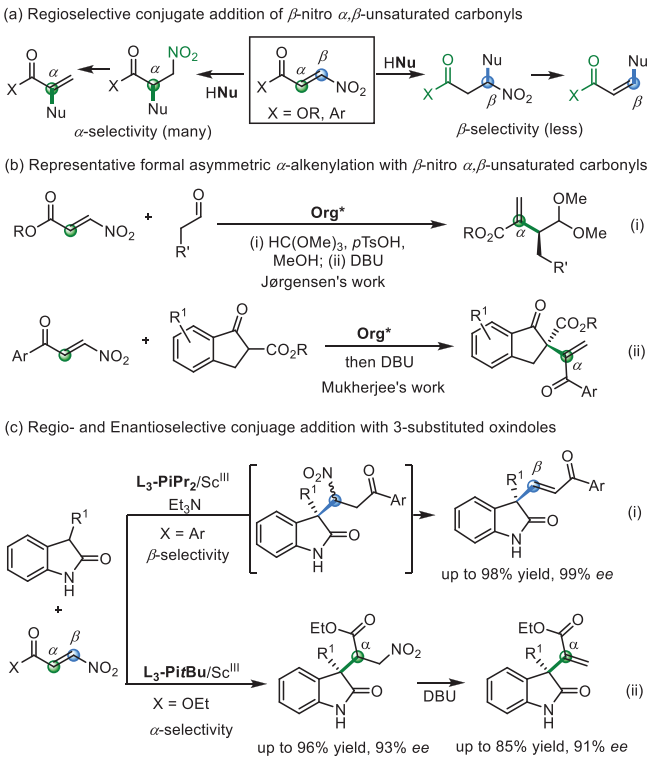
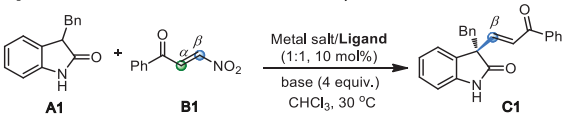


Scheme 1. Reaction about β-nitroenones and β-nitroacrylates.

Table 1
Optimizations of reaction conditions with β-nitroenone.[a]



A1 B1 C1

Entry	Metal salt	Ligand	Base	Yield (%)[b]	*ee* (%)[c]
1	$Mg(OTf)_2$	**L$_3$-PiPr$_2$**	Na_3PO_4	24	25
2	$Ni(OTf)_2$	**L$_3$-PiPr$_2$**	Na_3PO_4	19	3
3	$Yb(OTf)_3$	**L$_3$-PiPr$_2$**	Na_3PO_4	13	0
4	$Sc(OTf)_3$	**L$_3$-PiPr$_2$**	Na_3PO_4	81	94
5	$Sc(OTf)_3$	**L$_3$-PrPr$_2$**	Na_3PO_4	56	67
6	$Sc(OTf)_3$	**L$_3$-RaPr$_2$**	Na_3PO_4	61	74
7	$Sc(OTf)_3$	**L$_3$-PiEt$_2$**	Na_3PO_4	72	76
8	$Sc(OTf)_3$	**L$_3$-PiMe$_2$**	Na_3PO_4	44	55
9	$Sc(OTf)_3$	**L$_3$-PiPr$_2$**	$NaHCO_3$	76	94
10	$Sc(OTf)_3$	**L$_3$-PiPr$_2$**	Et_3N	96	98
11[d]	$Sc(OTf)_3$	**L$_3$-PiPr$_2$**	Et_3N	97	98
12[d,e]	$Sc(OTf)_3$	**L$_3$-PiPr$_2$**	Et_3N	81	97
13	$Sc(OTf)_3$	**L$_3$-PiPr$_2$**	–	16	83

[a] Unless otherwise noted, all reactions were carried out with metal salt/**Ligand** (1:1, 10 mol%), base (4.0 equiv.), **A1** (0.10 mmol) and **B1** (0.15 mmol) in $CHCl_3$ (1.0 mL) at 30 °C in air for 24 h.
[b] Isolated yields.
[c] Determined by HPLC on a chiral stationary phase.
[d] At 30 °C for 4 h.
[e] $Sc(OTf)_3$/**L$_3$-PiPr$_2$** (1:1, 5 mol%).

ordinating with chiral *N,N′*-dioxide ligand **L$_3$-PiPr$_2$** (Fig. 1) which was derived from L-pipecolic acid, were investigated in the presence of Na_3PO_4 as a base in $CHCl_3$ at 30 °C (see Supporting information for details). It was found that $Mg(OTf)_2$, $Ni(OTf)_2$, and $Yb(OTf)_3$ could promote the reaction while $Sc(OTf)_3$ was more effective to yield the formal alkenylation product (*E*)-**C1** *via* β-selective conjugate addition/nitro-elimination in 81% yield and 94% *ee* (entries 1–4), with different regio-selectivity from Mukherjee's report (Scheme 1b, ii). For ligand skeletons (see Supporting information for details), it seemed that both the amino acid backbone

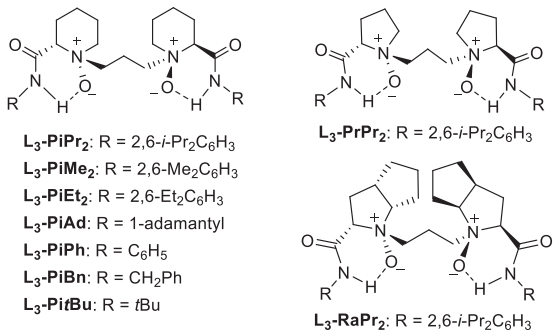


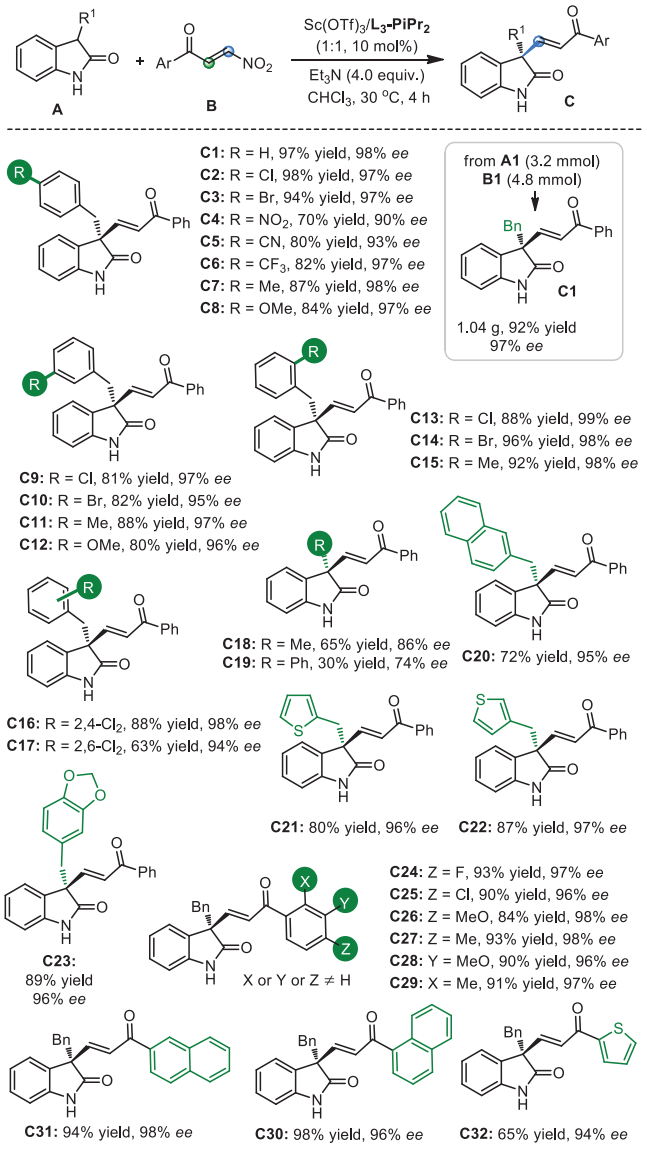
Fig. 1. Chiral *N,N′*-dioxides used.

and the steric hindrance of the amide subunits had dramatically influence on the reactivity and enantioselectivity. The ligand of **L$_3$-PrPr$_2$** (L-proline based one) and **L$_3$-RaPr$_2$** (L-ramipril based one) gave inferior results than **L$_3$-PiPr$_2$** (entries 4–6). Decreasing the steric hindrance of the 2,6-disubstituted amide from *i*Pr to Et or Me group led to dropped reactivity and enantioselectivity (entry 4 *vs.* entries 7 and 8). Bases affected the reactivity a little with slight change of the enantioselectivity (entries 9–11; see Supporting information for details). When the reaction was carried out with Et_3N instead of Na_3PO_4, 97% yield with 98% *ee* was obtained after 4 h (entry 11). At 5 mol% of catalyst loading, the enantioselectivity of the reaction could be maintained but the yield dropped a little (entry 12). If without a base, only 16% yield was obtained (entry 13).

With the optimized reaction conditions in hand (Table 1, entry 11), the substrate scope was evaluated (Scheme 2). Substituents at *para*-position of the benzyl group had little effect on the reactivity and enantioselectivity, regardless of the electron-donating groups or electron-withdrawing groups (70%–98% yields with 90%–98% *ee*, **C2**-**C8**) except for 4-NO_2-Bn (**C4**; 70% yield with 90% *ee*). Substituents at *ortho*-position or *meta*-position gave almost the same excellent enantioselectivities (80%–96% yields, 95%–99% *ee*, **C9**-**C16** and **C23**), but the 2,6-dichlorobenzyl-substituted **C17** was isolated in slightly lower yield (63%) with 94% *ee*. In particular, 3-substituted oxindoles, including methyl, 2-naphthylmethyl, 2-thienylmethyl, or 3-thienylmethyl, all were efficient nucleophiles to deliver the corresponding quaternary oxindole derivatives (**C18**, **C20**-**C22**) in 65%–87% yields with 86%–97% *ee*. In comparison, 3-phenyl substituted one afforded lower yield and *ee* value (**C19**, 30% yield and 74% *ee*). The absolute configuration of the product **C9** (CCDC: 2117166) was determined to be (*R,E*) by single-crystal X-ray diffraction analysis (for details, see Supporting information).

Different aryl β-nitroenones with diverse electronic and steric nature could be tolerated to generate the desired alkenylated products **C24**-**C29** in high yields with excellent *ee* values (84%–93% yields, 96%–98% *ee*). β-Nitroenones bearing 2-naphthyl, 1-naphthyl or 2-thienyl group, gave the products **C30**-**C32** in moderate to high yields with good enantioselectivities (65%–98% yields, 94%–98% *ee*). A scale up synthesis of internal vinyl substituted oxindole **C1** was accessible without obvious influence on the reactivity and enantioselectivity (92% yield and 97% *ee*).

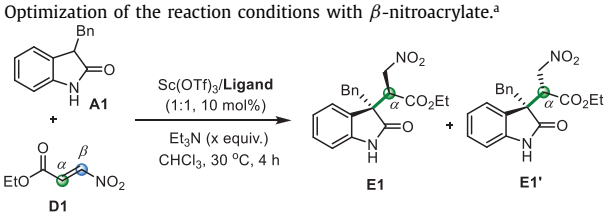
To investigate the influence of pull-substitution on the regioselectivity of dissymmetrical pull-pull alkenes, we tested the reaction between 3-substituted oxindole **A1** and β-nitroacrylate **D1**. However, as shown in Table 2, under the above optimal condition using **L$_3$-PiPr$_2$**/$Sc(OTf)_3$ and Et_3N, α-selective addition product **E1** and its diastereoisomer **E1′** were observed in good yield but with dramatically dropped enantioselectivity (Table 2, entry 1). The use of other *N,N′*-dioxide ligands (see Supporting information for details), such as **L$_3$-PrPr$_2$** and **L$_3$-RaPr$_2$**, gave nearly



Scheme 2. Substrate scopes of the reaction with β-selective addition. Unless otherwise noted, all reactions were carried out with Sc(OTf)$_3$/**L$_3$-PiPr$_2$** (1:1, 10 mol%), Et$_3$N (4.0 equiv.), **A** (0.10 mmol) and **B** (0.15 mmol) in $CHCl_3$ (1.0 mL) at 30 °C for 4 h. Yield was determined by Isolated yields. *ee* was determined by HPLC on a chiral stationary phase.

equal amount of diastereoisomers in poor enantioselectivity (entries 2 and 3). Interestingly, when less steric aniline-based **L$_3$-PiPh**, or phenylmethanamine-based **L$_3$-PiBn** used (Fig. 1), better enantioselectivity for every product was observed (entries 4 and 5). When aliphatic amine-based ligands employed, the yields of major product **E1** increased obviously (entries 6 and 7). Especially, the tertbutyl amine-based ligand **L$_3$-Pi*t*Bu** gave the product **E1** in 58% yield with 87% *ee*, and the product **E1′** in 23% yield with 82% *ee* (entry 7). Less amount of base is beneficial to the formation of α-selective product **E1** (entries 8 and 9), which could be isolated in 75% yield with 93% *ee* with 0.1 equiv. of Et$_3$N after prolonged reaction time at 0 °C (see Supporting information for details), and the minor diastereoisomer **E1′** was also obtained in 21% yield with 92% *ee* (entry 9). While if without base, the reaction did not occur and in the absence of DBU the nitro-group maintained in the products without elimination. The formal alkenylation products *via* elimination of nitro-group in the presence of DBU could be afforded without obvious erosion of the enantioselectivity from

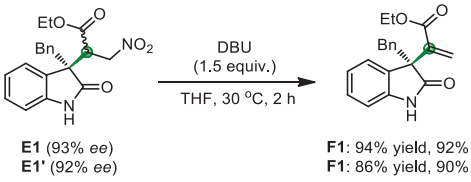
Table 2
Optimization of the reaction conditions with β-nitroacrylate.[a]



Entry	Ligand	E1′		E1	
		Yield (%)[b]	*ee* (%)[c]	Yield (%)[b]	*ee* (%)[c]
1	**L$_3$-PiPr$_2$**	37	30	52	36
2	**L$_3$-PrPr$_2$**	47	9	41	9
3	**L$_3$-RaPr$_2$**	46	10	43	8
4	**L$_3$-PiPh**	46	81	40	44
5	**L$_3$-PiBn**	36	70	41	68
6	**L$_3$-RiAd**	31	73	59	67
7	**L$_3$-Pi*t*Bu**	23	82	58	87
8[d]	**L$_3$-Pi*t*Bu**	24	88	76	90
9[e]	**L$_3$-Pi*t*Bu**	23 (21)[f]	92 (92)	76 (75)[f]	93 (93)

[a] Unless otherwise noted, all reactions were carried out with Sc(OTf)$_3$/**Ligand** (1:1, 10 mol%), Et$_3$N (4.0 equiv.), **A1** (0.10 mmol) and **D1** (0.15 mmol) in $CHCl_3$ (1.0 mL) at 30 °C in air for 4 h.
[b] Yield was determined by ^{1}H NMR experiments using $CHBr_2CHBr_2$ as an internal standard.
[c] Determined by HPLC or SFC on a chiral stationary phase.
[d] Sc(OTf)$_3$/**L$_3$-Pi*t*Bu** (1:1.1, 10 mol%), Et$_3$N (0.1 equiv.) in $CHCl_3$ (1.5 mL) at 30 °C in air for 4 h.
[e] Sc(OTf)$_3$/**L$_3$-Pi*t*Bu** (1:1.1, 10 mol%), Et$_3$N (0.1 equiv.) in $CHCl_3$ (1.5 mL) at 0 °C in air for 12 h.
[f] Isolated yields.

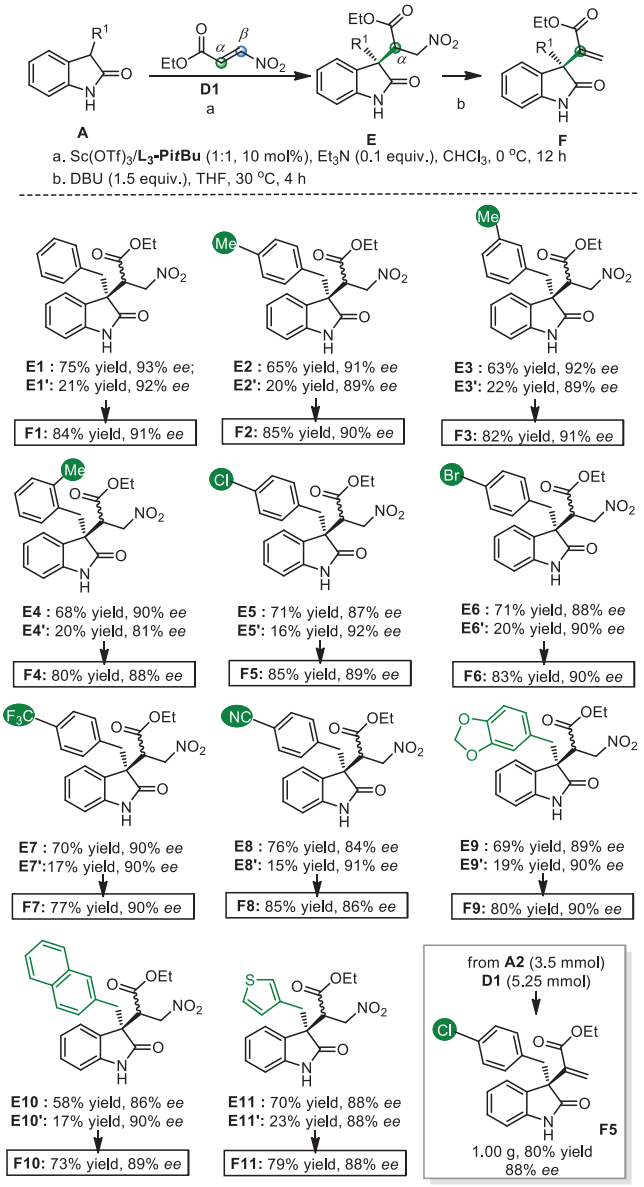
both α-selective addition product **E1** and **E1′** (Eq. 1), yielding the same formal alkenylation product **F1** with identical absolute configuration which showed that the diastereoselectivity raised from the α-position of nitroacrylate.



(1)

Having the optimal reaction conditions in hand (Table 2, entry 9), the scope of N–H 3-substituted oxindoles was evaluated (Scheme 3). Both electron-rich and electron-poor substituents at the phenyl group of 3-benzyl oxindoles were tolerated, furnishing the α-selective conjugate addition products **E2**-**E9** as the major products (63%–76% yield with 84%–92% *ee*), and the adducts **E2′**-**E9′** as the minor (15%–22% yield, 81%–92% *ee*). The electron-rich substituents gave slightly lower yield of α-selective adducts compared with the electron-withdrawing ones. 2-Naphthylmethyl oxindole was also tolerable, even though a moderate yield was obtained (58% yield with 86% *ee* for **E10**, and 17% yield with 90% *ee* for **E10′**). For 3-thienylmethyl oxindole, the corresponding product **E11** was isolated in 70% yield with 88% *ee*. When DBU was added to the finished asymmetric catalytic reaction mixture, the nitro elimination *exo*-methylene oxindoles **F1**-**F11** could be obtained in 73%–85% yields with 86%–91% *ee*. A gram-scale synthesis of *exo*-methylene oxindole **F5** in 80% yield and 88% *ee* demonstrated the reliability of this method.

We performed calculation about the natural bond orbital charges computed at the M06–2X/Def2-SVP level to unravel the factors controlling the regioselectivity of β-nitroenone **B1** and β-nitroacrylate **D2** [53]. As shown in Fig. 2a, the β-carbons of both **B1** and **D2** have stronger positive charges which seem to be more



Scheme 3. Substsrates scopes of regio- and enantio-selective reactions of β-nitroacrylate. Unless otherwise noted, all reactions were carried out with Sc(OTf)$_3$/**L$_3$-PitBu** (1:1.1, 10 mol%), Et$_3$N (0.1 equiv.), **A** (0.10 mmol) and **D1** (0.15 mmol) in $CHCl_3$ (1.5 mL) at 0 °C in air for 12 h. For the synthesis of formal alkenylation product **F**, DBU (1.5 equiv.) was added after the conjugate addition process finished. Yield was determined by isolated yields. *ee* was determined by HPLC or SFC on a chiral stationary phase.

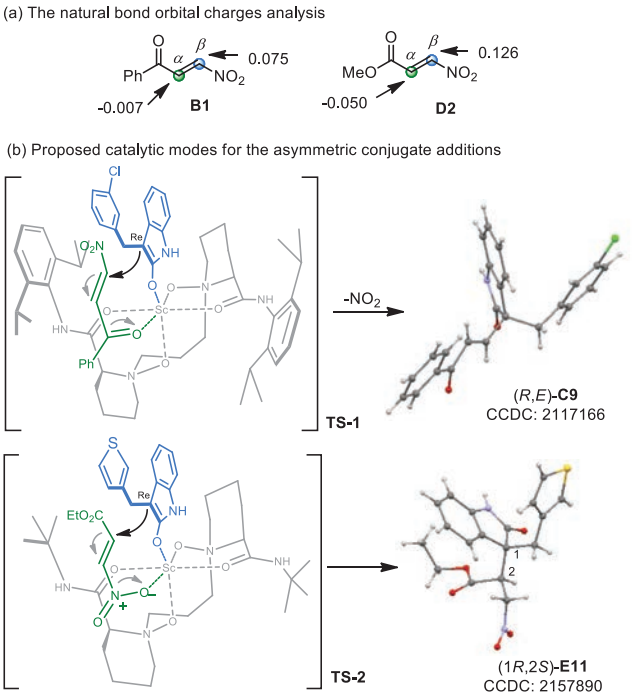


Fig. 2. Mechanism consideration.

likely to undergo nucleophilic attack, while the latter is inconsistent with the experimental results. It implies that it is difficult to evaluate the regioselectivity of dissymmetrical pull-pull alkenes in nucleophilic addition simply relying on natural bond polarizations, and the regioselectivity relies on entangled factors involving the interaction with the catalyst, steric hindrance, stability of the intermediates, and others. We rationalized that the reversal of regioselectivity in the addition reaction of β-nitroenone **B1** with β-nitroacrylate **D1** might be due to the selective coordination of the three kinds of pull groups, following the precedence as benzoyl > nitro > ester with the scandium catalyst. The Lewis acid activations occur at the benzoyl group of β-nitroenone **B1**, while at the nitro group of β-nitroacrylate **D1**, respectively, lowing the LUMO energy of the Michael acceptors to benefit the β-selective conjugate addition and α-selective conjugate addition, respectively.

The absolute configurations of the two products were determined *via* X-ray crystal analysis (for details, see Supporting information, pages 12–16), which showed the same stereoselectivity at the quaternary carbon center, indicating the same facial selectivity for the nucleophilic addition of the 3-substituted oxindoles. Based on the general catalytic behaviors of chiral Lewis acids of *N,N′*-dioxide ligands [48–52], we proposed a plausible possible catalytic modes to rationalize the regio- and enantioselectivity of the reaction. The four oxygens of *N,N′*-dioxide bond to the scandium center to form the chiral Lewis acid catalyst. As shown in the transition state **TS-1** of Fig. 2b, the benzoyl group of β-nitroenone **B1** coordinates to the metal center at the front site and the enolized oxindole bonds at the top site, leaving its *Re*-face toward the β-nitroenone. The *Re*-face of the Michael acceptor is blocked by the left amide subunit of the ligand, undergoing the addition with its *Si*-face to yield the β-selective addition intermediate. Upon elimination of the nitro group the formal alkenylation product (*R,E*)-**C9** was obtained as the major product. Similarly, when β-nitroacrylate **D1** is used as the Michael acceptor, the nitro group prefers to coordinate to the scandium center over ester group, and as shown in **TS-2** the enolized oxindole follows the similar coordination to the above **TS-1**. Thus, the α-selective adduct (1*R*,2*S*)-**E11** is generated as the major isomer. The supplementary crystallographic data of **E11** (CCDC: 2157890) can be obtained free of charge from The Cambridge Crystallographic Data centre.

To sum up, a highly efficient asymmetric Michael reaction of β-nitro α,β-unsaturated carbonyls with N–H 3-substituted oxindoles was realized by using chiral *N,N′*-dioxide/Sc(OTf)$_3$ complexes. The installation of internal or terminal vinyl substitution at the C3-position of oxindole was accessible *via* β-selective conjugate addition of β-nitroenones, and α-selective conjugate addition of β-nitroacrylates, respectively. The selective coordination of the scandium catalyst with pull-group of nitroolefins might account for the reversal of regioselectivity. Our protocols provided a useful route for the construction of functionalized quaternary oxindoles.

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.2022.05.001.

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