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Chiral *N,N′*-Dioxide/Lanthanide(III) Complex Catalyzed Asymmetric Bisvinylogous Mukaiyama Aldol Reactions

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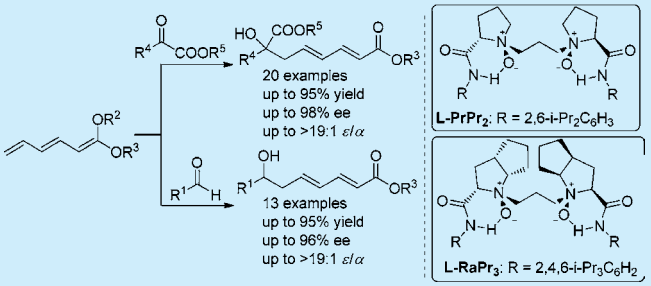
Supporting Information

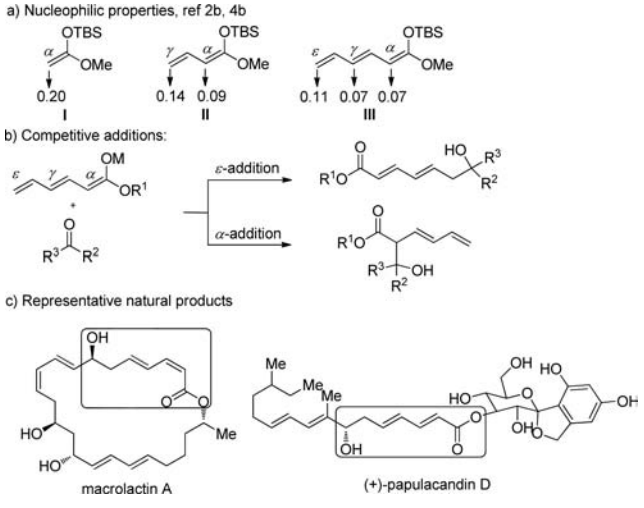
ABSTRACT: Chiral *N,N′*-dioxide/Y(OTf)$_3$ and Sc(OTf)$_3$ complexes have been developed as efficient catalysts for the bisvinylogous Mukaiyama aldol reaction of silyl ketene acetal with *α*-ketoesters and aldehydes, respectively. The catalytic systems were highly *ε*-selective, and the substrate scope was wide. The corresponding *ε*-hydroxy-*α,β,γ,δ*-unsaturated esters were obtained in up to 95% yield and 98% ee.



The vinylogous Mukaiyama aldol reactions (VMAR) are widely used in the synthesis of bioactive molecules. In particular, they provide a rapid assembly of polyketide segments with the advantage of avoiding extensive functional group manipulations as well as protecting group shuffling.

In the catalytic asymmetric field, reactions involving vinylogous silyl ketene acetal have been developed very well, and various catalytic systems have been reported.[1−3] However, for the reaction involving double-vinylogous silyl ketene acetal, only limited examples were reported.[4−6,9c] The difficulties lie in the weaker nucleophilicity of substrate and the competitive addition at the *α* and *ε* positions (Scheme 1a,b). List applied the disulfonimide as catalyst to promote the asymmetric Mukaiyama aldol reaction of **I**,[5a] **II**, and **III**,[5b] and moderate to good yields and ee were obtained. For the regioselectivity, the *γ*/*α* ratios in the reaction of **II** were as high as >40:1; however, *ε*/*α* ratios in the reaction of **III** varied from 0.6:1 to 9:1. Later, Curti developed a bisphosphoramide/SiCl$_4$ catalytic system for the hypervinylogous Mukaiyama aldol reactions (HVMAR) of furan-based silyloxypolyenes and highly unsaturated 2-silyloxyindoles with aldehydes.[6] Since the *ε*-hydroxy-*α,β,γ,δ*-unsaturated carbonyl structure exists in many biologically active molecules,[7] such as macrolactin A,[8] colletotriene, and (+)-papulacandin D (Scheme 1c),[9] and also is a key intermediate in the synthesis of natural products,[10] the improvement of yield, ee, and especially the *ε*/*α* ratio for the bVMAR of simple ester-derived silyl ketene acetal is required. In addition, other carbonyl compounds, like ketones, have not appeared in the asymmetric bVMAR. Therefore, developing new efficient catalystic systems for the asymmetric bVMAR with aldehydes as well as other carbonyl compounds is highly meaningful. Herein, we report our efforts in developing highly efficient chiral *N,N′*-dioxide/lanthanide(III) catalytic systems for the asymmetric bVMAR of silyl ketene acetal with *α*-ketoesters as well as aldehydes. Optically active *ε*-hydroxy-*α,β,γ,δ*-unsaturated carbonyl compounds were obtained in high yields, ee, and *ε*/*α* ratios under mild reaction conditions.

Scheme 1. (a) Calculated Nucleophilic Properties of Nucleophiles for the Mukaiyama Aldol Reaction; (b) Regioselectivity in the bVMAR; (c) Selected Examples of *ε*-Hydroxy-*α,β,γ,δ*-unsaturated Carbonyl



In the initial investigation, the reaction between silyl ketene acetal **1a** and *α*-ketoester **2a** was selected as a model reaction to optimize the reaction conditions. When a series of metal salts complexing with *N,N′*-dioxide **L−PrPr$_2$** were detected in THF at 35 °C, Sc(OTf)$_3$ gave only 10% yield, 30% ee, and 2:1 *ε*/*α* (Table 1, entry 1). Yb(OTf)$_3$ delivered 17% yield,

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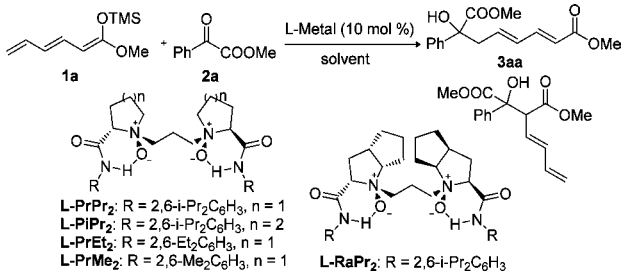
Table 1. Optimization of the Reaction Conditions[a]



entry	ligand	metal	solvent	yield[b] (%)	ee[c] (%)	ε/α[d]
1	**L−PrPr$_2$**	Sc(OTf)$_3$	THF	10	30	2:1
2	**L−PrPr$_2$**	Yb(OTf)$_3$	THF	17	58	4:1
3	**L−PrPr$_2$**	Y(OTf)$_3$	THF	31	67	>19:1
4	**L−RaPr$_2$**	Y(OTf)$_3$	THF	20	66	12:1
5	**L−PiPr$_2$**	Y(OTf)$_3$	THF	21	0	6:1
6	**L−PrEt$_2$**	Y(OTf)$_3$	THF	31	65	9:1
7	**L−PrMe$_2$**	Y(OTf)$_3$	THF	23	51	6:1
8	**L−PrPr$_2$**	Y(OTf)$_3$	THF	71	89	>19:1
9[e]	**L−PrPr$_2$**	Y(OTf)$_3$	THF	66	93	16:1
10[e,f]	**L−PrPr$_2$**	Y(OTf)$_3$	THF	84	93	>19:1

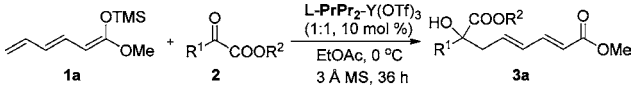
[a]Unless specified, all reactions were performed with L-metal (10 mol %, 1:1), **1a** (0.15 mmol), **2a** (0.10 mmol) in THF (0.5 mL) at 35 °C for 18 h. [b]Isolated yield of ε product. [c]Determined by HPLC analysis. [d]Determined by integration of the ^{1}H NMR spectrum of the crude products. [e]Reaction time was 36 h at 0 °C. [f]3 Å MS (20 mg) was added.

58% ee, and 4:1 ε/α (Table 1, entry 2). Surprisingly, Y(OTf)$_3$ dramatically improved the ratio of ε/α to >19:1 though the yield and ee were still low (31% yield, 67% ee, Table 1, entry 3). The amino acid backbone of the ligand was then probed. L-Proline derived **L−PrPr$_2$** was superior to L-ramipril derived **L−RaPr$_2$** (Table 1, entry 4) and L-pipecolic acid derived **L−PiPr$_2$** (Table 1, entry 5). The survey of the steric hindrance of *ortho*-substituents on the aniline ring of the *N*,*N*′-dioxide showed that the steric hindrance is crucial for chemical selectivity.

Decreasing the steric hindrance from 2,6-diisopropyl to 2,6-diethyl and 2,6-dimethyl, the ratio of ε/α was decreased sharply from >19:1 to 9:1 and 6:1 (Table 1, entry 3 vs entries 6 and 7). The yield and ee improved greatly with the ratio of ε/α maintained when the reaction was performed in EtOAc (71% yield, 89% ee, and >19:1 ε/α Table 1, entry 8). The ee value could be further increased to 93% when the reaction temperature was lowered to 0 °C with the yield and ε/α decreased slightly (Table 1, entry 9). Delightedly, when 3 Å molecular sieves were added to the system, **3aa** was obtained in 84% yield with 93% ee and >19:1 ε/α (Table 1, entry 10). Thus, the optimal conditions were set as a combination of **L−PrPr$_2$**/Y(OTf)$_3$ (1:1, 10 mol %) and 3 Å molecular sieves (20 mg) as additives in EtOAc (0.5 mL) at 0 °C.

With the optimized reaction conditions in hand, the scope of α-ketoesters **2** was explored by reaction with **1a** (Table 2). α-Ketoesters with aromatic, heteroaromatic, or aliphatic groups were all suitable substrates. Reactions of aromatic α-ketoesters with no matter electron-donating or electron-withdrawing substituents at different positions proceeded well, providing the corresponding products in 73%−95% yields with 92%−98% ee and >19:1 ε/α (Table 2, entries 1−15). Heteroaromatic **2p** and ring-condensed **2q** also reacted

Table 2. Substrate Scope of the bVMAR of Silyl Ketene Acetal with α-Ketoesters[a]



entry	R^1, R^2	3a	yield (%)[b]	ee (%)[c]	ε/α[d]
1	C$_6$H$_5$, Me	**3aa**	84	93	>19:1
2	3-MeC$_6$H$_4$, Me	**3ab**	77	98	>19:1
3	3-iPrC$_6$H$_4$, Me	**3ac**	80	97	>19:1
4	3-CH$_2$=CHC$_6$H$_4$,Me	**3ad**	73	97	>19:1
5	3-FC$_6$H$_4$, Me	**3ae**	76	97	>19:1
6	3-ClC$_6$H$_4$, Me	**3af**	89	95	>19:1
7	3-F$_3$CC$_6$H$_4$, Me	**3ag**	78	95	>19:1
8	4-MeOC$_6$H$_4$, Me	**3ah**	85	96	>19:1
9	4-tBuC$_6$H$_4$, Me	**3ai**	88	96	>19:1
10	4-PhC$_6$H$_4$, Me	**3aj**	84	96(*R*)	>19:1
11	4-CH$_2$=CHC$_6$H$_4$, Me	**3ak**	83	95	>19:1
12	4-FC$_6$H$_4$, Me	**3al**	76	93	>19:1
13	4-ClC$_6$H$_4$, Me	**3am**	81	95	>19:1
14	4-F$_3$CC$_6$H$_4$, Me	**3an**	82	92	>19:1
15	3,5-Me$_2$C$_6$H$_3$, Me	**3ao**	95	98	>19:1
16	2-thienyl, Et	**3ap**	75	95	>19:1
17	2-naphthyl, Me	**3aq**	88	98	>19:1
18	 , Et	**3ar**	75	95	>19:1
19	 , Et	**3as**	81	92	>19:1
20	c-hexyl, Me	**3at**	82	95	17:1

[a]Unless specified, all reactions were carried out with 10 mol % of **L−PrPr$_2$**/Y(OTf)$_3$ (1:1), **1a** (0.15 mmol), α-ketoesters (0.10 mmol), and 3 Å MS (20 mg) in ethyl acetate (0.5 mL) at 0 °C for 36 h. [b]Isolated yield of ε product. [c]Determined by HPLC analysis. [d]Determined by integration of the ^{1}H NMR spectrum of the crude products.

smoothly. The corresponding products **3ap** were obtained in 75% yield, 95% ee, and >19:1 ε/α and **3aq** in 88% yield, 98% ee, and >19:1 ε/α (Table 2, entries 16 and 17). In addition, linear or cyclic aliphatic α-ketoesters were also tolerant. The corresponding products **3ar−at** were obtained in 75−82% yields, 92−95% ee, and 17:1−>19:1 ε/α (Table 2, entries 18−20). The absolute configuration of product **3aj** was determined to be *R* by X-ray crystallographic analysis.

When the **L−PrPr$_2$**/Y(OTf)$_3$ catalytic system was extended to aldehydes, the bVMAR of silyl ketene acetal **1b** with benzaldehyde gave the corresponding **5ba** in only 24% yield albeit with 90% ee and >19:1 ε/α. Fortunately, by adjusting the catalytic system to **L−RaPr$_3$**/Sc(OTf)$_3$ (1:1.03, 10 mol %) with salicylic acid (20 mol %) as additive in pentyl hexanoate (0.5 mL) at −30 °C, 85% yield, 93% ee, and >19:1 ε/α could be obtained for **5ba** (see the optimization of the bVMAR with aldehydes in the SI). The reason for different central metals being efficient for the reactions of aldehydes and α-ketoesters might be that bidentate α-ketoesters coordinate better with atomic-radius-bigger Y(III), while monodentate aldehydes coordinate better with the atomic-radius-smaller Sc(III). For substituted benzaldehydes, electron-donating or electron-withdrawing substitutes on the

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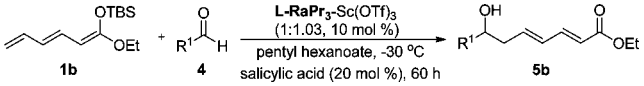
phenyl group were tolerated well, providing the corresponding products in 62−95% yields with 71−96% ee and 9:1−>19:1 ε/α (Table 3, entries 2−10). Generally, the benzaldehydes

Table 3. Substrate Scope of the bVMAR of Silyl Ketene Acetal with Aldehydes[a]



entry	R^1	5b	yield[b] (%)	ee[c] (%)	ε/α[d]
1	C_6H_5	**5ba**	85	93	>19:1
2	2-MeC_6H_4	**5bb**	75	87	17:1
3	2-$MeOC_6H_4$	**5bc**	88	96	17:1
4	2-BrC_6H_4	**5bd**	95	95(*S*)	>19:1
5	3-MeC_6H_4	**5be**	62	87	>19:1
6	3-$MeOC_6H_4$	**5bf**	68	89	>19:1
7	3-FC_6H_4	**5bg**	91	96	>19:1
8	3-BrC_6H_4	**5bh**	84	79	9:1
9	4-PhC_6H_4	**5bi**	81	77	18:1
10	4-BrC_6H_4	**5bj**	90	71	>19:1
11	1-naphthyl	**5bk**	89	91	>19:1
12		**5bl**	86	67	14:1
13		**5bm**	81	42	12:1

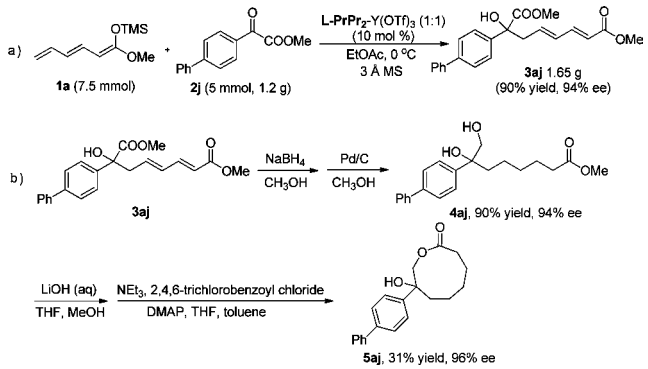
[a]Unless specified, all reactions were carried out with 10 mol % of **L−RaPr$_3$**/Sc(OTf)$_3$ (1:1.03), **1b** (0.15 mmol), aldehydes (0.10 mmol), and salicylic acid (20 mol %) in pentyl hexanoate (0.5 mL) at −30 °C for 60 h. [b]Isolated yield of ε product. [c]Determined by HPLC analysis. [d]Determined by integration of the ^{1}H NMR spectrum of the crude products.

with an electron-withdrawing substitutent led to no better yield than the ones with electron-donating substituents. Ring-condensed 1-naphthaldehyde **4k** was also suitable, and the corresponding product **5bk** was afforded in 89% yield, 91% ee, and >19:1 ε/α (Table 3, entry 11). For the aliphatic aldehyde **4m**, the product **5bm** was obtained in 81% yield and 42% ee with 12:1 ε/α (Table 3, entry 13). The absolute configuration of **5bd** was determined to be *S* by ^{1}H NMR analysis of the corresponding Mosher ester (see the Supporting Information).

To show the utility of the current method, a gram-scale synthesis of **3aj** was carried out. Pleasingly, by treatment of 5 mmol of **2j** and 7.5 mmol of **1a** in the presence of an **L−PrPr$_2$**/Y(OTf)$_3$ catalytic system, 1.65 g (90% isolated yield) of **3aj** with 94% ee was obtained (Scheme 2a). In addition, the highly enantiomerically enriched **3aj** can be easily converted into **5aj** (Scheme 2b).[5b]

To gain insight into the reaction mechanism, the nonlinear effect (NLE)[11] for the relationship between the enantiomeric excess of ligand **L−PrPr$_2$** and the product **3aa** was

Scheme 2. (a) Asymmetric Bisvinylogous Mukaiyama Aldol Reaction on a Gram Scale; (b) Transformation of 3aj to 5aj



investigated. A clear linear effect was observed, which suggested that the monomeric complex functioned as the most active and effective catalyst (Figure 1). Based on our

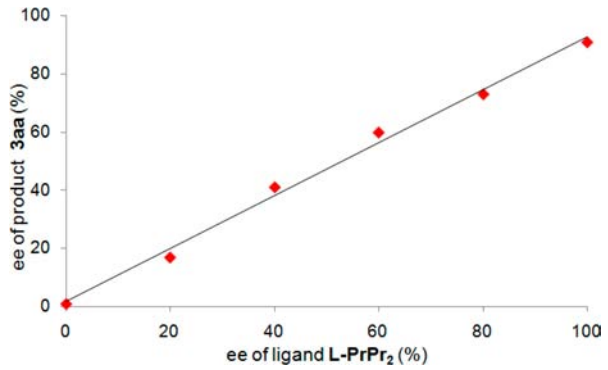


Figure 1. Investigation of nonlinear effect.

previous work[12] and the determination of the absolute configuration of product **3aj**, a possible transition state was proposed in Figure 2. The four oxygens of *N*,*N*′-dioxide **L−PrPr$_2$** and the bidentate α-ketoester **2j** coordinate with Y^{III} to form six-membered chelate rings. The *Re*-face of α-ketoester is shielded by the neighboring amide group of the ligand, and silyl ketene acetal **1a** attacks from the *Si*-face to form the product with *R* configuration.

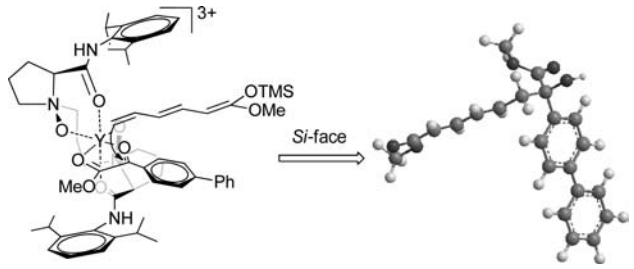


Figure 2. Proposed activation model and the absolute configuration of **3aj**.

In summary, we have developed a highly efficient catalyst system of **L−PrPr$_2$**/Y(OTf)$_3$ for the asymmetric bVMAR of α-ketoesters as well as **L−RaPr$_3$**/Sc(OTf)$_3$ for the reaction of silyl ketene acetal with aldehydes. The ε-selectivity was excellent, and the enantioselectivities were good to excellent.

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The utility of the methodology is highlighted by the gram-scale synthesis and the useful conversions. Further studies of chiral *N,N′*-dioxide/metal complexes catalyzing other reactions are ongoing.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b03470.

Experimental details, analytical data (NMR, HPLC, ESI-HRMS, and absolute configuration for for compounds **3aj** and **6bd** (PDF)

X-ray data for compound **3aj** (CIF)

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Notes

The authors declare no competing financial interest.

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