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Enantioselective Addition of Dialkylzincs to Aldehydes Catalyzed by (−)-MITH**

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Abstract: An effective catalytic system that imparts high enantioselectivity has been disclosed for the synthesis of optically active alcohols, which may undergo further chemical transformations. The enantioselective alkylation of aldehydes with dialkylzincs to afford the corresponding optically active alcohols with excellent enantioselectvities has been achieved in the presence of 0.1–0.5 mol% of the camphor-derived chiral ligand (−)-2-*exo*-morpholinoisobornane-10-thiol (MITH) (**1**) at room temperature or at 0°C.

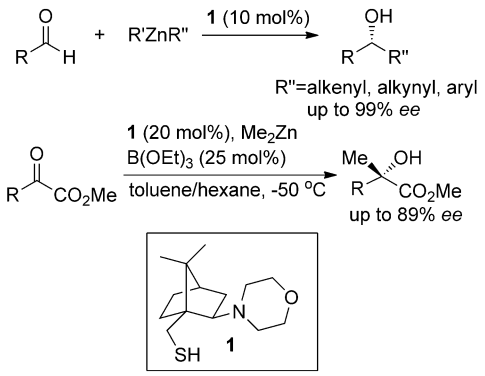
Keywords: alcohols · aldehydes · homogeneous catalysis · organozincs · enantioselective addition

Introduction

Enantioselective addition reactions of organozincs to carbonyl compounds or their imino derivatives,[1] in the presence of chiral catalysts to afford chiral alcohols or amines, have attracted the attention of synthetic organic chemists owing to their mild reaction conditions, high functional-group tolerance, and low-toxicity of zinc metal. The concomitant construction of a C–C bond and a new chiral center in the reaction course leads to its wide application in the synthesis of optically active alcohols that will undergo further elaboration to natural products and biologically active components.[2] Although notable examples on the development of a more effective ligand system for the addition reaction of dialkylzinc,[3] vinylzinc,[4] arylzinc,[3i,m,5] and alkynylzinc substrates[6] to carbonyl compounds have been reported, an efficient catalytic system for the highly enantioselective addition of dimethylzinc to aldehydes is sparse.[7] The development of a methodology for the enantioselective addition of dimethylzinc to carbonyl compounds remains challenging, because of the relatively lower reactivity of dimethylzinc. In this regard, chiral metal complexes have been recently reported to effect this transformation.[8]

Results and Discussion

As part of our endeavors to develop camphor-derived chiral ligands for catalytic enantioselective reactions,[9] (−)-2-*exo*-morpholinoisobornane-10-thiol (MITH) (**1**) has been demonstrated to be an effective promoter for the enantioselective addition of arylzinc,[9d] alkenylzinc,[9e] and alkynylzinc[9g] to aldehydes, thereby affording the corresponding optically active alcohols with excellent enantioselectvities (up to > 99% *ee*; Scheme 1). Ligand **1** was also employed as a chiral



Scheme 1. Enantioselective addition of organozincs to carbonyl compounds catalyzed by ligand **1**.

promoter in the enantioselective addition of dimethylzinc to α-ketoesters, thus providing the corresponding hydroxy esters that bear quaternary chiral centers with good yields and *ee*.[9f] Encouraged by these results, we anticipated that ligand **1** could impart high catalytic activity and a high level of enantioselectivity in the enantioselective addition of organozinc reagents to aldehydes. Herein, we describe our findings in the catalytic enantioselective addition reactions of dialkylzincs to aldehydes.

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[**] MITH = (−)-2-*exo*-morpholinoisobornane-10-thiol

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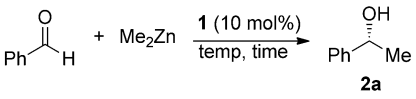
The enantioselective addition of dimethylzinc to benzaldehyde, catalyzed by 10 mol% of ligand **1**, was initially tested at 0°C. The effect that the amount of dimethylzinc used had on the enantioinduction was investigated and using two equivalents of dimethylzinc gave good yields and enantioselectivities (Table 1, entries 1–4). The enantioselective reaction conducted at room temperature furnished the optically active alcohol **2a** in good yield, with comparable enantioselectivity, and was completed in a shorter reaction time than that of a reaction at 0°C (Table 1, entry 5). Reactions that were carried out at lower temperature (Table 1, entry 6), at lower concentration (Table 1, entry 7) or in toluene (Table 1, entry 8), afforded no improvement in yields and enantioselectivities, respectively. The reaction conditions specified (Table 1, entry 5) were applied for further studies of the reaction parameters.

Table 1. Optimization of the enantioselective addition of Me_2Zn to benzaldehyde.

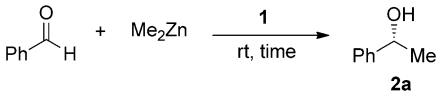


Entry	Me_2Zn [equiv][a]	T [°C]	t [h]	Yield [%][b]	*ee* [%][c]
1	1.5	0	72	58	89
2	2.0	0	48	88	94
3	3.0	0	20	84	95
4	5.0	0	18	89	95
5	2.0	25	6	82	93
6	2.0	10	48	82	94
7[d]	2.0	0	96	80	94
8[e]	2.0	0	48	70	89

[a] A 0.73 M solution of dimethylzinc in hexanes was used, the concentration was titrated according to the literature procedure.[10] [b] Yield of isolated product after column chromatography. [c] Determination by chiral HPLC. [d] The reaction mixture was diluted to half the concentration with hexanes. [e] A 1.0 M solution of dimethylzinc in toluene was used.

Subsequently, the effect that changing the amount of ligand **1** had on the enantioselective reaction between dimethylzinc and benzaldehyde was investigated (Table 2). Interestingly, the enantioselectivity did not vary as the amount of ligand decreased in the reaction, however, longer reaction times or more dimethylzinc were required to give reasonable yields for a lower loading of catalyst. The reaction rate was enhanced as more dimethylzinc was used (Table 2, cf. entry 1 vs 2; entry 6 vs 7). Given the efficiency and atom economy of the reaction, the reaction conditions with three equivalents of dimethylzinc in the presence of 0.5 mol% of ligand **1** (Table 2, entry 5) were applied to study the scope of the catalytic system with different aldehydes, and the results are summarized in Table 3. Generally, good yields and *ee* values were obtained with 0.5 mol% of chiral ligand **1**, except in the cases of *para*-CF_3-benzaldehyde (Table 3, entry 5), 2-furaldehyde and 3-furaldehyde (Table 3, entries 11 and 12), and cinnamaldehydes (Table 3, entries 15 and 16). Furthermore, 2 mol% of ligand **1** is required to provide good yields and enantioselectivities for 4-tolylbenzaldehyde and 1-naphthaldehyde (Table 3, entries 3 and 13). While the relationship of the enantiopurity of chiral ligand and that of the product is usually assumed to be linear, the convex deviation, positive nonlinear effect ((+)-NLE), also known as asymmetric amplification, was observed from the enantioselective addition of dimethylzinc to aldehyde in the presence of 2 mol% of ligand **1** (Figure 1).[11] Such effects arose from the reversible interchange of the coexisting homochiral and heterochiral zinc–amino thiolates. The homochiral complex dissociated to form a monomeric enantioselective zinc complex in this reaction, thus giving rise to the high enantioinduction, whereas the heterochiral one remained the same and inactive.

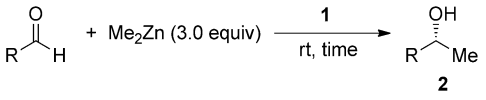
Table 2. Effect of catalyst loading on the enantioselective addition of Me_2Zn to benzaldehyde.



Entry	**1** [mol%]	Me_2Zn [equiv][a]	t [h]	Yield [%][b]	*ee* [%][c]
1	5.0	2.0	24	85	93
2	5.0	5.0	6	89	94
3	2.0	2.0	24	88	94
4	1.0	2.0	96	82	93
5	0.5	3.0	48	89	94
6	0.1	3.0	96	68	92
7	0.1	5.0	72	88	93

[a] A 0.73 M solution of dimethylzinc in hexanes was used.[10] [b] Yield of isolated product. [c] Determined by chiral HPLC.

Table 3. Enantioselective addition of Me_2Zn to aldehydes catalyzed by ligand **1**.[a]



Entry	R	**1** [mol%]	t [h]	Yield [%][b]	*ee* [%][c]
1	2-Tol	0.5	48	82 (**2b**)	90
2	3-Tol	0.5	48	80 (**2c**)	90
3	4-Tol	2.0	48	89 (**2d**)	96
4	4-MeO-Ph	0.5	72	82 (**2e**)	85
5	4-CF_3-Ph	2.0	48	91 (**2f**)	78
6	2-Cl-Ph	0.5	48	83 (**2g**)	84
7	3-Cl-Ph	0.5	48	86 (**2h**)	90
8	4-Cl-Ph	0.5	48	77 (**2i**)	93
9	4-Br-Ph	0.5	48	70 (**2j**)	89
10	2-Thienyl	0.5	48	82 (**2k**)	92
11	2-Furyl	0.5	48	77 (**2l**)	78
12	3-Furyl	0.5	48	82 (**2m**)	83
13	1-Naphthyl	2.0	48	90 (**2n**)	93
14	2-Naphthyl	0.5	48	86 (**2o**)	92
15	Cinnamyl	0.5	48	95 (**2p**)	64
16	2-Me-Cinnamyl	0.5	72	65 (**2q**)	83
17	hydrocinnamyl	0.5	48	83 (**2r**)	42
18	cyclohexyl	0.5	48	70 (**2s**)	86

[a] A 0.73 M solution of dimethylzinc in hexanes was used.[10] [b] Yield of isolated product. [c] Determined by chiral HPLC.

After obtaining good results from the enantioselective addition of dimethylzinc to a variety of arylaldehydes, the

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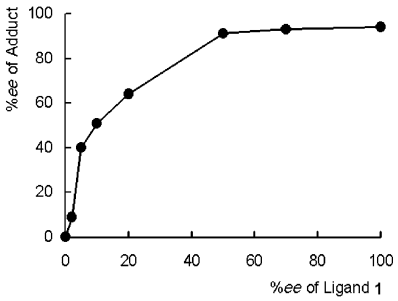
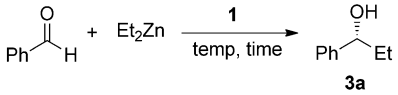


Figure 1. Nonlinear effect of ligand **1** in the enantioselective addition of dimethylzinc to benzaldehyde.[12]

enantioselective addition of diethylzinc[3] to benzaldehyde was investigated. Excellent enantioselectivities (91–97%) and yields were obtained with 0.1–5 mol% of ligand **1** (Table 4). In the presence of only 0.5 mol% of ligand **1**, the reaction was completed in 30 min and furnished chiral alcohol **3a** in high *ee* at room temperature, whereas a longer

Table 4. Effect of catalyst loading on the enantioselective addition of diethylzinc to benzaldehyde.



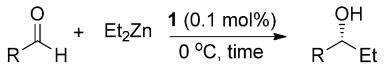
Entry	**1** [mol%]	Et_2Zn [equiv][a]	*T* [°C]	*t* [h]	Yield [%][b]	*ee* [%][c]
1	5.0	2.0	0	28	95	97
2	1.0	2.0	0	24	90	97
3	0.5	1.5	rt	0.5	94	95
4	0.5	1.5	0	24	95	96
5	0.1	1.5	rt	4	95	91
6	0.1	1.5	0	24	95	96
7	0.05	1.5	0	48	56	20

[a] A 1.0 M solution of dimethylzinc in hexanes was used. [b] Yield of isolated product. [c] Determined by chiral HPLC.

reaction time is necessary for the reaction conducted at 0°C (Table 4, entry 3 vs 4). Further studies on the reactions in the presence of 0.1 mol% of ligand **1** indicated that higher enantioinduction was observed at 0°C (Table 4, entry 5 vs 6). The product **3a** was obtained in very low *ee* (20%) when the ethylation reaction was conducted with 0.05 mol% of ligand **1** (Table 4, entry 7). Therefore, the reaction conditions specified in entry 6 were applied to study the scope of this reaction (Table 5).

Excellent enatioselectivities (92–99%) of the corresponding secondary alcohols were obtained from the enantioselective addition of diethylzinc to aromatic aldehydes that bear various substituents (Table 5, entries 1–10), except in the case of *para*-chlorobenzaldehyde (Table 5, entry 8). The enantioselective reaction with cinnamaldehyde afforded chiral alcohol **3l** in good yield (95%) but with lower enantioselectivity (71% *ee*; Table 5, entry 11) than that of its α-substituted congener (77% yield and 90% *ee*; Table 5, entry 12).

Table 5. Enantioselective addition of diethylzinc to aldehydes catalyzed by ligand **1**.[a]



Entry	R	*t* [h]	Yield [%][b]	*ee* [%][c]
1	2-Tol	24	95 (**3b**)	94
2	3-Tol	24	94 (**3c**)	94
3	4-Tol	24	95 (**3d**)	97
4	4-MeO-Ph	48	94 (**3e**)	93
5	4-CF_3-Ph	24	96 (**3f**)	97
6	2-Cl-Ph	24	88 (**3g**)	99
7	3-Cl-Ph	24	92 (**3h**)	96
8	4-Cl-Ph	48	80 (**3i**)	86
9	4-Br-Ph	24	85 (**3j**)	96
10	2-Naphthyl	24	94 (**3k**)	95
11	Cinnamyl	24	95 (**3l**)	71
12	2-Me-Cinnamyl	48	77 (**3m**)	90
13	hydrocinnamyl	24	83 (**3n**)	64
14	cyclohexyl	24	56 (**3o**)	82

[a] A 1.0 M solution of dimethylzinc in hexanes was used. [b] Yields of isolated product. [c] Determined by chiral HPLC.

In contrast to our previous studies, alkylation of aromatic aldehydes catalyzed by ligand **1** requires a much lower catalyst loading. Presumably, the structure of the alkyl zincate is different from that in previous studies. However, the real reason remains to be understood. The mechanism of the enantioselective addition of dialkylzinc to aldehydes in the presence of a catalytic amount of β-amino alcohols[1b,13] and β-amino thiols[14] has been reported. We have proposed structure **4** as the major transition state in the catalytic addition reaction (Figure 2). Formation of a six-membered cyclic

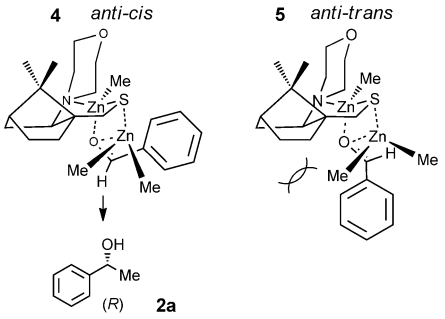


Figure 2. Proposed reaction transition state of the enantioselective addition of Me_2Zn to benzaldehyde catalyzed by ligand **1**.

ring might occur by the coordination of sulfur and nitrogen atoms with one zinc atom. In our case, the *anti-cis* structure (**4**), rather than the *anti-trans* structure (**5**) that is the usual transition state, has exhibited a minimized steric repulsion between the phenyl ring and the bornane skeleton, and therefore the transfer of the *anti* methyl group from the *Re*-face of the benzaldehyde gives rise to the observed (*R*)-1-phenyl-1-ethanol (**2a**).

Conclusions

In conclusion, the preparation of highly enantioenriched chiral secondary alcohols through the enantioselective addition of dialkylzinc to a family of aromatic aldehydes catalyzed by ligand **1** has been demonstrated. The presented catalytic system shows high catalytic activity and enantioselectivity, and, thus a low catalyst loading is required. For methylation, 0.5 mol% of ligand **1** was required, whereas for ethylation, as low as 0.1 mol% of ligand **1** was required.

Experimental Section

A stock solution of ligand **1** was prepared by mixing ligand **1** (12.8 mg, 0.05 mmol) and a dimethylzinc solution (5.0 mL, 3.7 mmol, 0.73 M in hexanes) at room temperature for 5 min. The stock solution (0.5 mL) was added to a flame-dried flask and the mixture was stirred at room temperature for 5 min before the addition of the aldehyde (1.0 mmol). After stirring at room temperature for 48 h, aqueous ammonium chloride (2 mL, 1 N solution) was added to quench the reaction. The mixture was then neutralized with 1 N HCl (aq), and the organic solution was extracted with ethyl acetate (3×10 mL). The combined organic extracts were dried over anhydrous Na_2SO_4, and concentrated to give the crude product, which was purified by column chromatography to yield the corresponding adducts. The *ee* was determined by HPLC on the chiral stationary phase.

Acknowledgements

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[12] The investigation was conducted with 2 mol% of ligand **1** and 2 equivalents of dimethylzinc in hexanes.

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[14] For an example: C. L. Gibson, *Tetrahedron: Asymmetry* **1999**, *10*, 1551–1561.

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