



Stereoselective Synthesis of 1,2-Amino Alcohols by Addition of Organocuprate•BF₃ Complexes and Organolithium Reagents to an α -Silyloxyaldimine Derived from (*S*)-Ethyl Lactate

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Abstract: 1,2-Amino alcohols were synthesized by addition of organocuprate•BF₃ **2** (R=*n*-Bu, *n*-octyl, Me, phenethyl, Ph), organocopper•BF₃ **3** (R=Me, *n*-Bu), and organolithiums (R=*n*-Bu, *n*-octyl, Me, phenethyl, Ph) to an α -silyloxyaldimine **1** derived from commercially available (*S*)-ethyl lactate. The reactions with organocuprate•BF₃ **2** and organocopper•BF₃ **3** led to the *anti* isomers almost exclusively (*anti:syn*=98:<2 for R=*n*-Bu, *n*-octyl, Me, phenethyl and *anti:syn*=95:5 for R=Ph, 52-93% isolated yields) and the *syn* isomers could be obtained with high stereoselectivities by using organolithiums (*anti:syn*=10:90~20:80, 41-71% isolated yields). Copyright © 1996 Elsevier Science Ltd

INTRODUCTION

1,2-Amino alcohols have been important building blocks in the synthesis of various natural products¹ and bicyclic amidines and guanidines.² In addition, 1,2-amino alcohols have been used as chiral sources in many asymmetric induction reactions involving metal reagents.³ However, stereoselective synthesis of 1,2-amino alcohols by nucleophilic addition to imines has been rather troublesome especially with enolizable imines.⁴ Based on our report about the effectiveness of organocuprate•BF₃ complexes in the reaction of various aldimines,⁵ we decided to apply the reagents to chiral aldimines. Here we report stereoselective synthesis of 1,2-amino alcohols by the nucleophilic addition of organocuprate•BF₃ complexes and organolithium reagents to an α -silyloxyimine which could easily be prepared from commercially available (*S*)-ethyl lactate.

RESULTS AND DISCUSSION

Reactions of organocuprate•BF₃ complexes to α -silyloxyaldimine **1** proceeded smoothly to give *anti* isomers of 1,2-amino alcohols stereoselectively in high isolated yields after deprotection with tetrabutylammonium fluoride (TBAF) as summarized in Table 1 (entries 1, 4, 8, 10 and 12). Table 1 shows wide applicability of the reaction. Notable is the fact that fairly unreactive Ph₂CuLi•BF₃⁶ is also effective. The high levels of diastereoselectivities were not nucleophile dependent and only a small amount of the corresponding *syn* isomer was obtained in the case of Ph₂CuLi•BF₃ (entry 12). BF₃ played an essential role for the addition, and

organocuprates themselves did not give any products in the absence of BF_3 (entries 7 and 14).⁵ The formation of organocuprate $\cdot\text{BF}_3$ complex prior to the reaction was also essential since the addition of organocuprate to the solution of **1** and BF_3 was unproductive. Organocopper $\cdot\text{BF}_3$ complexes were also effective if two equivalents of these complexes were used (entries 3 and 6: with one equivalent of the complex, the yield was lower).⁷ Interestingly, reactions of **1** with organolithium reagents proceeded to give *syn* isomers predominantly (*anti* : *syn* = 10:90–20:80) (entries 2, 5, 9, 11 and 13).⁸ Thus each diastereomer could be prepared selectively only by changing the choice of organometallic reagents.

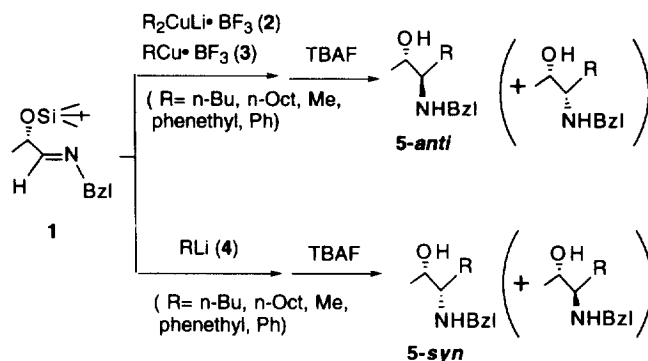


Table 1. Stereoselectivities of Addition of Organometallic Reagents to 1

entry	nucleophile	product	<i>anti</i> : <i>syn</i>	yield (%)	$J_{\text{H}_a/\text{H}_b}$ (Hz) <i>anti</i> / <i>syn</i>
1	$\text{Me}_2\text{CuLi}\cdot\text{BF}_3$	5 a	>98:<2	78	3.4
2	MeLi	5 a	18:82	41	6.4
3	$\text{MeCu}\cdot\text{BF}_3$ (2.0 equiv.)	5 a	>98:<2	93	3.4
4	$n\text{-Bu}_2\text{CuLi}\cdot\text{BF}_3$	5 b	>98:<2	80	3.4
5	$n\text{-BuLi}$	5 b	19:81	43	7.8
6	$n\text{-BuCu}\cdot\text{BF}_3$ (2.0 equiv.)	5 b	>98:<2	52	3.4
7	$n\text{-Bu}_2\text{CuLi}$	5 b	-	0	-
8	$(n\text{-Octyl})_2\text{CuLi}\cdot\text{BF}_3$	5 c	>98:<2	85	3.4
9	$(n\text{-Octyl})\text{Li}$	5 c	11:89	71	4.4
10	$(\text{PhCH}_2\text{CH}_2)_2\text{CuLi}\cdot\text{BF}_3$	5 d	>98:<2	75	3.6
11	$\text{PhCH}_2\text{CH}_2\text{Li}$	5 d	10:90	54	4.4
12	$\text{Ph}_2\text{CuLi}\cdot\text{BF}_3$	5 e	95:5	58	4.4
13	PhLi	5 e	20:80	67	8.3
14	Ph_2CuLi	5 e	-	0	-

The relative stereochemistries of the 1,2-amino alcohols of **5** [**a**] R=Me, **b**] R=n-Bu, **e**] R=Ph] were assigned according to an empirical rule, i.e., coupling constants of *anti* isomers ($J=3.4-4.4\text{Hz}$) are smaller than those of *syn* isomers ($J=6.4-8.3\text{Hz}$, Table 1).⁹ However, in the case of **5** [**c**] R=n-octyl, **d**] R=phenethyl], the coupling constants were very similar, i.e., 3.4-3.6Hz (*anti*) and 4.4Hz (*syn*), and one cannot be confident on the assignment of relative stereochemistry.

In order to confirm the assignment, oxazolidin-2-ones **6**^{10,11} were prepared from corresponding 1,2-amino alcohols (**5**) as shown in Scheme 2. When H^a was irradiated, NOE was observed for H^b of **6b-anti-(4R)** (5.8%) and **6c-anti-(4R)** (7.4%) but not for **6b-syn-(4S)**. Also, X-ray crystallographic structural analysis of **5e-anti-(1R)** was carried out, supporting the assignment of relative stereochemistry by the coupling constant (Figure 1). Although these results are not contradictory to the empirical rule, we should be very careful for the assignment when substituents are larger or bulkier compared to common examples.

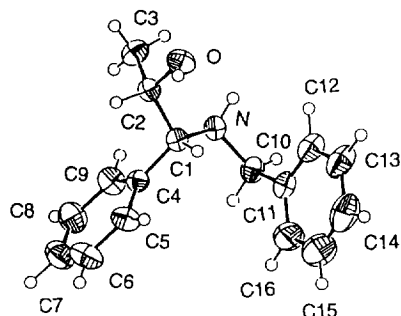
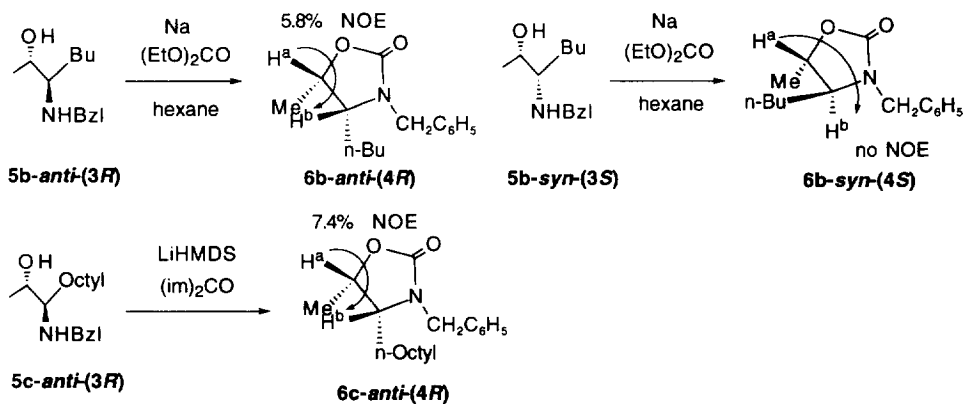


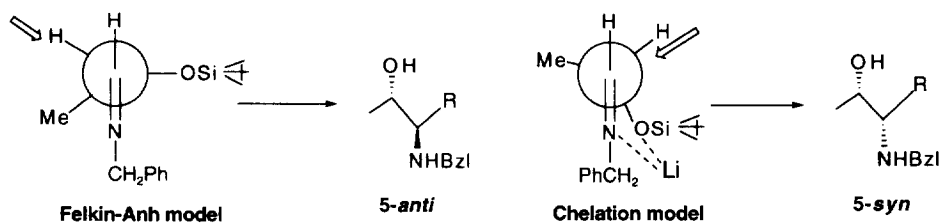
Figure 1. X-ray crystal structure of **5e-anti**.



Scheme 2

The *syn* addition of organolithium reagents can be explained by the chelation model and the *anti* selectivity with organocuprate•BF₃ complexes can be accounted for by the Felkin-Anh model (Scheme 3). The quite high

anti selectivities are surprising in light of the fact that the addition of $\text{BuCu}\cdot\text{BF}_3$ and $\text{Bu}_2\text{CuLi}\cdot\text{BF}_3$ to $\text{Ph}(\text{Me})\text{CH}-\text{C}(\text{H})=\text{NPr}$ gives a rather low diastereoselectivity (4:1).¹² The difference can be rationalized as follows. The Felkin-Anh conformer¹³ shown in Scheme 3 should play a predominant role in the reaction of **1** because of the electron-withdrawing effect of the oxygen atom and the bulkiness of the silyloxy group compared with the methyl group. In contrast, smaller difference of the steric and electronic effects between the phenyl and the methyl substituents in $\text{Ph}(\text{Me})\text{CH}-\text{C}(\text{H})=\text{NPr}$ may lead to several conformers responsible for the addition.



EXPERIMENTAL

¹H NMR spectra were recorded at 400MHz (JEOL EX400) in CDCl_3 in the presence of TMS as an internal standard. Melting points are uncorrected.

Preparation of **1**.

Aldimine **1** was prepared by the reaction of the corresponding silyloxyaldehyde¹⁴ with benzylamine in the presence of molecular sieves 4A in ether. The crude aldimine was purified by vacuum distillation (116-120 °C/0.7mmHg).

General Procedure for the Addition of Organocuprate $\cdot\text{BF}_3$ with an α -Silyloxyimine (**1**).

To a stirred solution of 5.04 mmol of RLi in dry THF (20 mL) at -30 °C was added 0.51g (2.67 mmol) of CuI over a period of 5 min and the reaction mixture was stirred for 20 min at -30 °C. After the solution was cooled to -78 °C and was stirred for 20 min, $\text{BF}_3\cdot\text{Et}_2\text{O}$ (0.33 mL, 2.67 mmol) was added dropwise over a period of 5 min. The solution was stirred for 15 min at the same temperature, then 0.75 mL (2.52 mmol) of imine **1** was added to the solution and the reaction mixture was allowed to warm to room temperature and stirred overnight. The reaction was quenched with 30 mL of aqueous NaOH (10%). After the mixture was filtered through Celite, the filtrate was extracted with ether (50 mL x 3). The combined organic layer was dried over anhydrous Na_2SO_4 . After filtration, the solvent was evaporated to give the *t*-butyldimethylsilyl protected product which was purified by TLC (*i*-PrNH₂:hexane=1:30).

To a stirred solution of *t*-butyldimethylsilyl protected amino alcohol containing a mixture of *anti* and *syn* isomers in THF (1.0 mL) was added TBAF in THF (1M, 0.46 mL, 0.46 mmol) at 0 °C and the reaction mixture was stirred for 2 h at 0 °C and at room temperature for 1 day. The mixture was treated with H₂O (30 mL) and was extracted with ether (50 mL x 3). The combined organic layer was dried over anhydrous Na_2SO_4 . After

filtration, the solvent was evaporated and the residue was chromatographed (TLC, ether/*i*-PrNH₂=100:1) to give 1,2-amino alcohol.

General Procedure for the Addition of Organolithium Reagents to 1.

To a stirred solution of the imine **1** (0.75ml, 2.52mmol) in dry hexane (20 mL) at -78 °C was added 2.77 mmol of RLi solution in hexane over a period of 5 min and the reaction mixture was allowed to warm to room temperature and stirred for one day, followed by treatment with 30 mL of aqueous NaOH (10%). The mixture was extracted with ether (50 mL x 3), and the combined organic layer was dried over anhydrous Na₂SO₄. After filtration, the solvent was evaporated and the residue was chromatographed (TLC, *i*-PrNH₂:hexane=1:40) to give *t*-butyldimethylsilyl protected product. Desilylation was carried out by similar procedures described above.

(2S,3R)-3-Benzylamino-2-heptanol (5b-anti). The product could be obtained pure by recrystallization (ether / hexane=1/1). mp 92.2-92.7 °C, ¹H NMR (CDCl₃) δ 0.90-1.11 (m, 3H), 1.07 (d, 3H, *J* = 6.4 Hz), 1.24-1.65 (m, 7H), 2.35-2.45 (m, 1H), 2.50-2.55 (m, 1H), 3.80 (s, 2H), 3.88 (dq, 1H, *J* = 3.4, 6.4 Hz), 7.20-7.40 (m, 5H). Anal. Calcd for C₁₄H₂₃NO: C, 75.97; H, 10.47; N, 6.33. Found: C, 75.83; H, 10.67; N, 6.24.

(2S,3S)-3-Benzylamino-2-heptanol (5b-syn). The product could be obtained pure for NOE spectra by separation with recycle HPLC (1,2-dichloroethane). ¹H NMR (CDCl₃) δ 0.87-0.94 (m, 3H), 1.18 (d, 3H, *J* = 6.4 Hz), 1.30-1.65 (m, 7H), 2.32-2.45 (m, 2H), 3.47 (dq, 1H, *J* = 7.8, 6.4 Hz), 3.71, 3.87 (ABq, 2H, *J* = 12.7 Hz), 7.25-7.40 (m, 5H). This was converted to oxazolidinone (**6b-syn**).

(1R,2S)-1-Benzylamino-1-phenyl-2-propanol (5e-anti). The product could be obtained pure by recrystallization (ether / hexane=1/1). mp 84.0-85.0 °C, ¹H NMR (CDCl₃) δ 0.98 (d, 3H, *J* = 6.4 Hz), 3.29 (d, 1H, *J* = 9.3 Hz), 3.54, 3.69 (ABq, 2H, *J* = 13.2 Hz), 3.74 (dq, 1H, *J* = 9.3, 6.4 Hz), 7.22-7.39 (m, 10H). Anal. Calcd for C₁₆H₁₉NO: C, 79.63; H, 7.94; N, 5.80. Found: C, 79.30; H, 7.94; N, 5.69.

(1S,2S)-1-Benzylamino-1-phenyl-2-propanol (5e-syn). The product could be obtained pure by recrystallization (ether / hexane=1/1). mp 77.5-79.0 °C, ¹H NMR (CDCl₃) δ 1.01 (d, 3H, *J* = 6.4 Hz), 3.65, 3.77 (ABq, 2H, *J* = 13.2 Hz), 3.70 (d, 1H, *J* = 4.4 Hz), 4.01 (dq, 1H, *J* = 4.4, 6.4 Hz), 7.23-7.39 (m, 10H). Anal. Calcd for C₁₆H₁₉NO: C, 79.63; H, 7.94; N, 5.80. Found: C, 79.26; H, 7.87; N, 5.64.

(2S,3R)-3-Benzylamino-2-butanol (5a-anti). The product could be obtained pure by recrystallization (ether / hexane=1/1). mp 84.0-85.0 °C, ¹H NMR (CDCl₃) δ 1.01 (d, 3H, *J* = 6.4 Hz), 1.10 (d, 3H, *J* = 6.4 Hz), 2.72 (dq, 1H, *J* = 3.4, 6.4 Hz), 3.81 (s, 2H), 3.84 (dq, 1H, *J* = 3.4, 6.4 Hz), 7.24-7.36 (m, 5H). Anal. Calcd for C₁₁H₁₇NO: C, 73.70; H, 9.56; N, 7.81. Found: C, 73.84; H, 9.61; N, 7.51.

(2S,3S)-3-Benzylamino-2-butanol (5a-syn). ¹H NMR (CDCl₃) δ 1.11 (d, 3H, *J* = 6.4 Hz), 1.18 (d, 3H, *J* = 6.4 Hz), 2.41 (dq, 1H, *J* = 8.3, 6.4 Hz), 3.34 (dq, 1H, *J* = 8.3, 6.4 Hz), 3.70, 3.94 (ABq, 2H, *J* = 13.2 Hz), 7.22-7.38 (m, 5H). HRMS(M+1) 180.1380 (calcd for C₁₁H₁₇NO 180.1388).

(2S,3R)-3-Benzylamino-2-undecanol (5c-anti). The product could be obtained pure by recrystallization (ether / hexane=1/1). mp 86.0-87.0 °C, ¹H NMR (CDCl₃) δ 0.88 (t, 3H, *J* = 6.8 Hz), 1.09 (d, 3H, *J* = 6.4 Hz), 1.10-1.50 (m, 14H), 2.52-2.56 (m, 1H), 3.81 (s, 2H), 3.87 (dq, 1H, *J* = 6.4, 3.4 Hz), 7.20-7.40 (m, 5H). Anal. Calcd for C₁₈H₃₁NO: C, 77.92; H, 11.26; N, 5.05. Found: C, 78.18; H, 11.24; N, 5.08.

(2S,3S)-3-Benzylamino-2-undecanol (5c-syn). ¹H NMR (CDCl₃) δ 0.89 (t, 3H, *J* = 6.8 Hz), 1.19 (d, 3H, *J* = 6.8 Hz), 1.10-1.60 (m, 14H), 2.36 (dt, 1H, *J* = 4.4, 6.8 Hz), 3.47 (dq, 1H, *J* = 4.4, 6.8 Hz), 3.72, 3.87 (ABq, 2H, *J* = 12.7 Hz), 7.21-7.38 (m, 5H). HRMS(M+1) 278.2466 (calcd for C₁₈H₃₁NO 278.2484).

(2S,3R)-3-Benzylamino-5-phenyl-2-pentanol (5d-anti). The product could be obtained pure by recrystallization (ether / hexane=1/1). mp 87.0-87.5 °C, ¹H NMR (CDCl₃) δ 1.11 (d, 3H, *J* = 6.4 Hz), 1.68-1.80 (m, 2H), 2.50-2.75 (m, 3H), 3.76, 3.80 (ABq, 2H, *J* = 13.2 Hz), 3.91 (dq, 1H, *J* = 3.6, 6.4 Hz), 7.10-7.35 (m, 10H). Anal. Calcd for C₁₈H₂₃NO: C, 80.25; H, 8.61; N, 5.20. Found: C, 80.48; H, 8.69; N, 5.25.

(2S,3S)-3-Benzylamino-5-phenyl-2-pentanol (5d-syn). ¹H NMR (CDCl₃) δ 1.21 (d, 3H, *J* = 6.4 Hz), 1.70-1.81 (m, 1H), 1.86-1.98 (m, 1H), 2.44 (dt, 1H, *J* = 4.4, 7.3 Hz), 2.62-2.75 (m, 2H), 3.57 (dq, 1H, *J* = 4.4, 6.4 Hz), 3.73, 3.86 (ABq, 2H, *J* = 12.7 Hz), 7.10-7.35 (m, 10H). HRMS(M) 269.1786 (calcd for C₁₈H₂₃NO 269.1780).

(4R,5S)-cis-3-Benzyl-4-*n*-butyl-5-methyl-1,3-oxazolidin-2-one (6b-anti). **6b-Anti** was prepared from **5b-anti** using diethyl carbonate and EtONa in hexane.¹⁰ The product was purified with recycle HPLC (1,2-dichloroethane). When H^a was irradiated, NOE was observed for H^b (5.8%) (Scheme 2). ¹H NMR (CDCl₃) δ 0.80 (t, 3H, *J* = 7.3 Hz), 1.26 (d, 3H, *J* = 6.3 Hz), 1.10-1.60 (m, 6H), 3.44 (dt, 1H, *J* = 3.4, 6.3 Hz), 3.98 (d, 1H, *J* = 15.6 Hz), 4.53 (quintet, 1H, *J* = 6.3 Hz), 4.73 (d, 1H, *J* = 15.6 Hz), 7.15-7.30 (m, 5H). HRMS(M) 247.1568 (calcd for C₁₅H₂₁NO₂ 247.1572).

(4S,5S)-trans-3-Benzyl-4-*n*-butyl-5-methyl-1,3-oxazolidin-2-one (6b-syn). **6b-Syn** was prepared from **5b-syn** using diethyl carbonate and EtONa in hexane.¹⁰ The product was purified with recycle HPLC (1,2-dichloroethane). When H^a was irradiated, NOE was not observed for H^b (Scheme 2). ¹H NMR (CDCl₃) δ 0.79 (t, 3H, *J* = 7.9 Hz), 1.07-1.55 (m, 6H), 1.23 (d, 3H, *J* = 5.9 Hz), 2.98-3.04 (m, 1H), 3.95 (d, 1H, *J* = 15.1 Hz), 4.18 (quintet, 1H, *J* = 5.9 Hz), 4.73 (d, 1H, *J* = 15.1 Hz), 7.05-7.30 (m, 5H). HRMS(M) 247.1572 (calcd for C₁₅H₂₁NO₂ 247.1572).

(4R,5S)-cis-3-Benzyl-5-methyl-4-*n*-octyl-1,3-oxazolidin-2-one (6c-anti). **6c-Anti** was prepared from **5c-anti** using lithium hexamethyldisilazide and carbonyl diimidazole.¹¹ The product was purified with recycle HPLC (1,2-dichloroethane). When H^a was irradiated, NOE was observed for H^b (7.4%) (Scheme 2). ¹H NMR (CDCl₃) δ 0.88 (t, 3H, *J* = 6.8 Hz), 1.33 (d, 3H, *J* = 6.6 Hz), 1.23-1.56 (m, 14H), 3.51 (dt, 1H, *J* = 3.4, 6.6 Hz), 4.05 (d, 1H, *J* = 15.6 Hz), 4.60 (quintet, 1H, *J* = 6.6 Hz), 4.80 (d, 1H, *J* =

15.6 Hz), 7.26-7.36 (m, 5H). HRMS(M) 303.2180 (calcd for C₁₉H₂₉NO₂ 303.2199).

Crystal Structure of **5e-anti**.

Crystallographic data for **5e-anti**: C₁₆H₁₉NO, FW =241.3, *orthorhombic*, space group *P2₁2₁2₁*, *a* = 13.662(2) Å, *b* = 18.822(3) Å, *c* = 5.261(1) Å, *V* = 1352.8(4) Å³, *Z* = 4, *D* cal = 1.18 g/cm³, μ = 0.40 cm⁻¹. A Crystal suitable for X-ray structure determination was mounted on a Mac Science MXC3 diffractometer and irradiated with graphite-monochromated Mo *K* α radiation (λ = 0.71073 Å) for data collection. Lattice parameters were determined by least-squares fitting of 31 reflections with 25° < 2 θ < 30°. Data were collected with the 2 θ / ω scan mode. The structure was solved by a direct method with a program, Monte Carlo-Multan.¹⁵ Refinement on *F* was carried out by full-matrix least-squares. All non-hydrogen atoms were refined with anisotropic thermal parameters. All hydrogen atoms could be found on a difference Fourier map; these coordinates were included in the refinement with isotropic thermal parameters. All the computations were carried out on a Titan-750 computer using the *crystal-G* program. The final cycle of full-matrix least-squares refinement was based on 1155 reflections [*F* > 3.00 σ (*F*)] and 221 variable parameters with *R*(*Rw*) = 0.039 (0.044). Atomic coordinates, bond distances and angles, and thermal parameters have been deposited at the Cambridge Crystallographic Data Center.

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