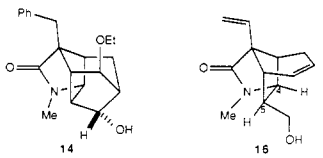
π-complexes A–D are considered as intermediates, which very much resemble the transition-state structures. Our results (Table I) show that (*E*)-**10** preferably cyclizes through A and (*Z*)-**10** favors the pathway through D. That A is more favorable than B can be understood by realizing that the atoms participating in the mechanism of five-membered-ring formation (boldface bonds in Scheme II) adopt a chair conformation in A vs a boat in B.[21,22] The same reasoning in comparing C and D gives the wrong answer, however. That D is preferred over C can be explained by invoking a severe steric interaction between the silyloxy function and the cyclohexene ring in C.

The *N*-acyliminium cyclization of the inseparable 70/30 *E*/*Z* mixture of **11** (BF_3·OEt_2, CH_2Cl_2, 20 °C, 5 min) gave a 70/30 mixture of the aldehydes **4** and **15**, respectively, showing that the stereospecificity is independent of the nature of the bridgehead substituent. Without purification, the aldehyde mixture was immediately reduced ($NaBH_4$, EtOH) to alcohols **16** and its C-5 epimer in 70% overall yield from **11**. Recrystallization of the latter mixture provided pure **16**[8] (mp 97–98 °C), which exhibited a singlet for H-4 in its ^1H NMR spectrum, proving the stereochemistry at C-5.[16] Definitive structural proof was obtained as follows. Treatment of **16** with iodine (Na_2CO_3, MeCN, 20 °C, 5 days) furnished tetracycle **2**[8] in 49% yield,[23] as a crystalline solid (mp 141–143 °C). This compund was subjected to a single-crystal X-ray diffraction study (Figure 1),[24] which nicely revealed the expected tetracyclic structure with an axial iodine substituent in a chair cyclohexane ring, and a boat-like tetrahydropyran ring.



In conclusion, we have developed an efficient route (nine steps from (*E*)-3,5-hexadien-1-ol) to the tetracyclic skeletal part of gelsemine. Our current studies are concerned with the introduction of the oxindole moiety[25] starting from **16**, and we hope to eventually accomplish the total synthesis of this intriguing alkaloid.

Acknowledgment. We thank K. Goubitz and D. Heijdenrijk of the Laboratory of Crystallography, University of Amsterdam, for the X-ray structural determination, C. Kruk and his staff for this help in obtaining and interpreting the NMR spectra, and Fang Ya for the purification of alcohol **16**. Use of the services and facilities of the Dutch CAOS/CAMM Center, under grant numbers SON-11-20-700 and STW-NCH-44.0703, is gratefully acknowledged.

Registry No. 1, 509-15-9; (±)-**2**, 115095-85-7; (±)-**5**, 115095-74-4; (±)-**8**, 115095-75-5; (±)-(*E*)-**9**, 115095-76-6; (±)-(*Z*)-**9**, 115095-77-7; (±)-(*E*)-**10**, 115095-78-8; (±)-(*Z*)-**10**, 115095-79-9; (±)-(*E*)-**11**, 115095-80-2; (±)-(*Z*)-**11**, 115095-81-3; (±)-**12**, 115095-82-4; (±)-**13**, 115182-33-7; (±)-**13** (diethyl acetal), 115095-83-5; (±)-**14**, 115095-86-8; (±)-**16**, 115095-84-6; (±)-5-*epi*-**16**, 115182-34-8; (*E*)-$HO(CH_2)_2CH{=}CHCH{=}CH_2$, 73670-87-8; *N*-methylmaleimide, 930-88-1.

(23) The remaining 51% consisted mainly of starting material and aldehyde **4**, apparently formed by means of iodine-mediated oxidation; the corresponding tetrahydrofuran was not found.

(24) Details of the X-ray study may be found in the supplementary material.

(25) (a) Fleming, I.; Loreto, M. A.; Wallace, I. H. M.; Michael, J. P. *J. Chem. Soc., Perkin Trans. 1* **1986**, 349. (b) Jones, K.; Thompson, M.; Wright C. *J. Chem. Soc., Chem. Commun.* **1986**, 115. (c) Abelman, M. M.; Oh, T.; Overman, L. E. *J. Org. Chem.* **1987**, *52*, 4130.

Supplementary Material Available: Spectral data of compounds **2**, **8**, (*E*)-**10**, (*Z*)-**10**, **12–14**, and **16** and details of the single-crystal X-ray structure determination of **2** (7 pages). Ordering information is given on any current masthead page.

Henk Hiemstra,* Robert Jan Vijn
W. Nico Speckamp*
Laboratory of Organic Chemistry
University of Amsterdam
Nieuwe Achtergracht 129
1018 WS Amsterdam, The Netherlands
Received March 3, 1988

Highly Diastereoselective Deprotonation and Substitution of Chiral 5,6-Dihydro-4*H*-1,2-oxazines

Summary: Deprotonation of the chiral 1,2-oxazine **1** by *n*-butyllithium provides a carbanion which reacts highly diastereoselectively with electrophiles affording the substituted 1,2-oxazines **2**. The overall substitution occurs under retention of configuration in most cases investigated. These remarkable results are in accord with recent ab initio calculations.

Sir: 5,6-Dihydro-4*H*-1,2-oxazines (herein abbreviated as 1,2-oxazines) are highly promising heterocyclic intermediates for the construction of polyfunctional compounds.[1] They are most easily prepared by [4 + 2] cycloaddition of nitrosoalkenes to (electron-rich) olefins.[2] C-4-substituted derivatives should be available by conversion of 1,2-oxazines to carbanions and subsequent reaction with appropriate electrophiles. This reaction sequence is well-known for oxime ethers[3] and the related isoxazolines.[4] Indeed, Shatzmiller has reported on the regiochemistry of the deprotonation of 5,6-dihydro-3-methyl-4*H*-1,2-oxazine.[5] To our best knowledge no other lithiated 1,2-oxazines have been studied. In this paper we disclose our results with the chiral 6-(trimethylsilyl)methyl-substituted 1,2-oxazine **1**,[1h] which demonstrate that deprotonation and reactions

(1) For first applications and leading references, see: (a) Gilchrist, T. L.; Lingham, D. A.; Roberts, T. G. *J. Chem. Soc., Chem. Commun.* **1979**, 1089. (b) Gilchrist, T. L.; Roberts, T. G. *J. Chem. Soc., Chem. Commun.* **1979**, 1090. (c) Faragher, R.; Gilchrist, T. L. *J. Chem. Soc., Perkin Trans. 1* **1979**, 249. (d) Oppolzer, W.; Bättig, K.; Hudlicky, T. *Tetrahedron* **1981**, *37*, 4359. (e) Ottenheijm, H. C.; Plate, R.; Noordik, J. H.; Herscheid, J. D. M. *J. Org. Chem.* **1982**, *47*, 2147. (f) Nakanishi, S.; Shirai, Y.; Takahashi, K.; Otsuji, Y. *Chem. Lett.* **1981**, 869. (g) Nakanishi, S.; Higuchi, M.; Flood, T. C. *J. Chem. Soc., Chem. Commun.* **1986**, 30. (h) Hippeli, C.; Reissig, H.-U. *Synthesis* **1987**, 77. (i) Chrystal, E. J. T.; Gilchrist, T. L.; Stretch, W. *J. Chem. Res. Synop.* **1987**, 180; *J. Chem. Res. Miniprint* **1987**, 1563. (j) Li, J. P.; Newlander, K. A.; Yellin, T. O. *Synthesis* **1988**, 73.

(2) See ref 1 and the following. (a) Review: Gilchrist, T. L. *Chem. Soc. Rev.* **1983**, *12*, 53. (b) Intramolecular cycloaddition: Denmark, S. E.; Dappen, M. S.; Sternberg, J. A. *J. Org. Chem.* **1984**, *49*, 4741.

(3) (a) Spencer, T. A.; Leong, C. W. *Tetrahedron Lett.* **1975**, 3889. (b) Fraser, R. R.; Dhawan, K. L. *J. Chem. Soc., Chem. Commun.* **1976**, 674. (c) Ensley, H. E.; Lohr, R. *Tetrahedron Lett.* **1978**, 1415. (e) Shatzmiller, S.; Lidor, R. *Synthesis* **1983**, 590.

(4) (a) Jäger, V.; Schwab, W. *Tetrahedron Lett.* **1978**, 3129. Grund, H.; Jäger, V. *Liebigs Ann. Chem.* **1980**, 80. Jäger, V.; Buss, V.; Schwab, W. *Liebigs Ann. Chem.* **1980**, 122. Schwab, W.; Jäger, V. *Angew. Chem., Int. Ed. Engl.* **1981**, *20*, 603. Jäger, V.; Müller, I.; Schohe, R.; Frey, M.; Ehrler, R.; Häfele, B.; Schröder, D. *Lect. Heterocycl. Chem.* **1985**, *8*, 79. For an extensive discussion of the structure and reactivity of isoxazoline anions, see: Ehrler, R. Dissertation, Würzburg, 1985. (b) Shatzmiller, S.; Shalom, E.; Lidor, R.; Tartkovski, E. *Liebigs Ann. Chem.* **1983**, 906. (c) Kozikowski, A. P.; Ghosh, A. K. *J. Org. Chem.* **1984**, *49*, 2762. (d) Annunziata, R.; Cinquini, M.; Cozzi, F.; Raimondi, L. *Tetrahedron* **1986**, *42*, 2129. (f) Curran, D. P.; Chao, J.-C. *J. Am. Chem. Soc.* **1987**, *109*, 3036.

(5) Lidor, R.; Shatzmiller, S. *J. Am. Chem. Soc.* **1981**, *103*, 5916. Shatzmiller, S.; Lidor, R.; Shalom, E. *Isr. J. Chem.* **1986**, *27*, 33.

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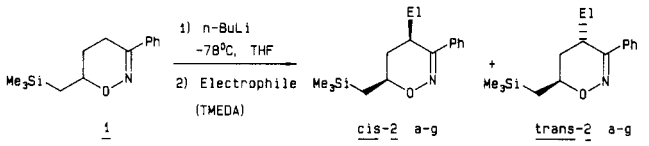
Table I. Reactions of Deprotonated 1,2-Oxazine 1 with Electrophiles[a]

entry	electrophile	El	product[b]	cis:trans[c]	yield,[d] %	mp, °C
a	D_2O	D	**2a**	>97:3	quant	42
b	MeI[e]	Me	**2b**	>97:3	58	118
c	allyl bromide[e]	allyl	**2c**	>97:3	83	52–56
d	acetone	Me_2COH	**2d**	>97:3	74	123–124
e	benzophenone	Ph_2COH	**2e**	>97:3	53	145–149
f	Me_2S_2	MeS	**2f**	70:30	85	90–92
g	acetaldehyde	MeHCOH	**2g**	50:50[f]	83	liq

[a] Conditions: 1 mmol of 1 in 10 mL of THF, 1.5 equiv of *n*-BuLi, 15 min, −78 °C, 3 equiv of electrophile, −78 °C (for entries d–g), −78 → 20 °C (for entry a), aqueous workup, purification by chromatography and/or recrystallization. [b] All products gave appropriate spectra and satisfactory elemental analyses. [c] According to high-field ^1H NMR spectroscopy of the *crude product*. [d] Yield of purified product. [e] Presence of 1 equiv of TMEDA during deprotonation and alkylation, −78 → 20 °C. [f] Four diastereomers in a ratio of approximately 1:1:1:1.

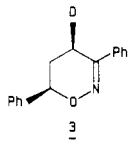
of the generated carbanion are highly stereoselective.

Addition of 1.5 equiv of *n*-butyllithium to **1** in tetrahydrofuran at −78 °C smoothly produces the corresponding carbanion.[6] Treatment of the red solution with D_2O gives a quantitative yield of the 4-deuteriated 1,2-oxazine **2a**.



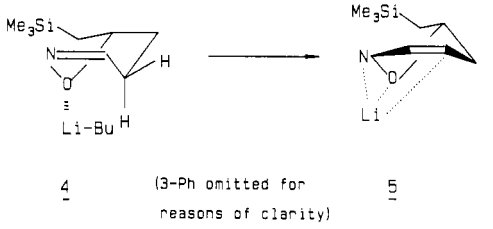
Surprisingly, only one of two diastereomers was formed. ^1H NMR data unambiguously reveal that the 6-(trimethylsilyl)methyl group and the deuterium in **2a** occupy pseudoequatorial positions in a half-chair conformation.[7] Thus D^+ is introduced only cis to the C-6 substituent.

Similarly, methyl iodide,[8] allyl bromide,[8] acetone, and benzophenone give exclusively one diastereomer (Table I, entries b–e). The NMR data at hand show that these electrophiles also afford 4,6-cis-substituted 1,2-oxazines.[9] With dimethyl disulfide as electrophile (entry f), *cis*-**2f** still predominates (cis:trans = 70:30), whereas acetaldehyde reacts unselectively, producing all four possible diastereomers in approximately equal quantities (entry g). The mixture of *cis*/*trans*-**2f** could be equilibrated with sodium methoxide in methanol (24 h, 20 °C)[10] providing a 20:80 cis:trans ratio.[11] As demonstrated by exclusive formation of **3** from the corresponding 1,2-oxazine, the stereoselective deuteriation is not restricted to **1**.[12]



Control experiments establish that only one of the two protons at C-4 is abstracted by the base. Thus, treatment of *cis*-**2a** with *n*-butyllithium and quench with D_2O leads to reisolation of apparently unchanged *cis*-**2a**. No trace of the double deuteriated material could be detected. In complete agreement with this result, *cis*-**2a** provides **1** or *cis*-**2d** after deprotonation and reaction with H_2O or acetone, respectively. The 4-methylated 1,2-oxazine *cis*-**2b** could not be deprotonated, even under more vigorous reaction conditions. These experiments unequivocally demonstrate that only the proton (or deuterium) located cis to the C-6 substituent is removed by *n*-butyllithium and that *the overall substitution occurs with retention of configuration.*

So far, we can only speculate with regard to the origins of the stereoselective deprotonation. Possibly, a boat conformation with the bulky 6-(trimethylsilyl)methyl group in a pseudoequatorial position fulfils most favorably the stereoelectronic requirements for the proton abstraction. Only in conformation **4** is the C–H bond to be broken coplanar with the p-orbitals of the C=N system. In addition, complexation of the incoming *n*-butyllithium by the 1,2-oxazine oxygen is achievable in **4**. Thus, the carbanion would directly accommodate structure **5** with interactions of Li^+ to oxygen, nitrogen, and C-4. A very similar ion pair for metalated oxime ethers has recently been suggested on the basis of ab initio calculations.[13]



Interpretation of the reaction between the metalated 1,2-oxazine and an electrophile is even more speculative. The ion pair **5** might be configurationally stable and the Li^+ cation in **5** could direct the attacking electrophile to the bottom face of the carbanion. Thus the S_E2 process occurs with retention, providing cis products **2** in most cases.[13,14] With electrophiles displaying lower affinity toward lithium (Me_2S_2) or being too reactive (MeCHO),

(6) We could not observe any addition product to the C=N bond. For addition of organometallics to this unit, see: Kolasa, T.; Sharma, S.; Miller, M. J. *Tetrahedron Lett.* **1987**, *28*, 4973 and references therein.

(7) ^1H NMR ($CDCl_3$, 400 MHz): δ 3.79 (dtd, J = 2, 7, 11 Hz, 6-H), 2.60 (tdd, $J_{H/D}$ = 2.5 Hz, J = 8, 11 Hz, 4-H), 2.10 (ddd, J = 2.5, 8, 13.5 Hz, 5-H_e), 1.78 (td, J = 11, 13.5 Hz, 5-H_a), 1.15 and 0.93 (2 dd, J = 7, 15 Hz, 6-CH_2), 0.10 (s, 9 H, $SiMe_3$).

(8) Alkyl halides as electrophiles require activation of the carbanion by tetramethylethylendiamine (TMEDA). Otherwise, no reaction occurs at −78 °C, or deprotonated **1** decomposes at higher temperature.

(9) Chemical shifts (δ) of 4-H and coupling constants (Hz) to 5-H_2: **2a**, 2.60/8, 11; **2b**, 2.99/7, 11; **2c**, 3.10/8, 11; **2d**, 3.20/9, 7.5; **2e**, 4.19/5, 10; *cis*-**2f** 3.96/10, 10; *trans*-**2f** 3.73/≈1.5, 4. For related heterocyclohexene systems and their NMR data, see: (a) Maier, M.; Schmidt, R. R. *Liebigs Ann. Chem.* **1985**, 2261. (b) Cook, M. J.; Desimoni, G. *Tetrahedron* **1971**, *27*, 257. (c) Sommer, S. *Chem. Lett.* **1977**, 583.

(10) Probably due to the lower acidity, 1,2-oxazine **2b** could not be isomerized under these conditions.

(11) It is possible that *trans*-**2f** is thermodynamically more stable for steric reasons, since the interaction of the 3-phenyl group and the pseudoequatorial 4-SMe group is avoided. In addition, the allylic effect might also contribute to the higher stability of *trans*-**2f**. For this stereoelectronic effect, see: Angerbauer, R.; Schmidt, R. R. *Carbohydr. Res.* **1981**, *89*, 193 and references therein.

(12) So far compounds with 6-OR (R = alkyl or $SiMe_3$) could not be metalated.

(13) (a) Glaser, R.; Streitwieser, A., Jr. *J. Am. Chem. Soc.* **1987**, *109*, 1258. (b) Glaser, R.; Streitwieser, A., Jr. *Pure Appl. Chem.* **1988**, *60*, 195.

(14) For comparable investigations see ref 3b and: Lyle, R. E.; Saavedra, J. E; Lyle, G. G.; Fribush, H. M.; Marshall, J. L.; Lijinsky, W.; Singer, G. M. *Tetrahedron Lett.* **1976**, 4431. Blagg, J.; Davies, S. G. *Tetrahedron* **1987**, *43*, 4463. For very interesting results with respect to stereoselective substitutions of chiral metalated formamidines, see: (a) Meyers, A. I.; Dickman, D. A. *J. Am. Chem. Soc.* **1987**, *109*, 1263 and earlier work of this group. (b) Gawley, R. E. *J. Am. Chem. Soc.* **1987**, *109*, 1265.

the steering effect of the cation is less pronounced and mixtures of cis/trans isomers result. However, these speculations need to be consolidated by further experiments including other electrophiles and under variation of reaction conditions.

The easy availability of diverse 4-substituted 1,2-oxazines such as **2** with defined stereochemistry has important synthetic consequences in light of the ring-opening reactions of these heterocycles.[15]

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Registry No. **1**, 109925-98-6; *cis*-**2a**, 115117-93-6; *trans*-**2a**, 115117-94-7; *cis*-**2b**, 115117-95-8; *trans*-**2b**, 115117-96-9; *cis*-**2c**, 115117-97-0; *trans*-**2c**, 115117-98-1; *cis*-**2d**, 115117-99-2; *trans*-**2d**, 115118-00-8; *cis*-**2e**, 115118-01-9; *trans*-**2e**, 115118-02-0; *cis*-**2f**, 115118-03-1; *trans*-**2f**, 115118-04-2; *cis*-**2g** (diastereomer 1), 115118-05-3; *cis*-**2g** (diastereomer 2), 115183-53-4; *trans*-**2g** (diastereomer 1), 115183-54-5; *trans*-**2g** (diastereomer 2), 115183-55-6.

(15) Products **2b** and **2d** could be ring opened analogously to reactions described in ref 1h. For related ring opening reactions of isoxazolines, see ref 4a.

Claudia Hippeli, Hans-Ulrich Reissig*
Institut für Organische Chemie der Technischen Hochschule Darmstadt
Petersenstr. 22, D-6100 Darmstadt, FRG
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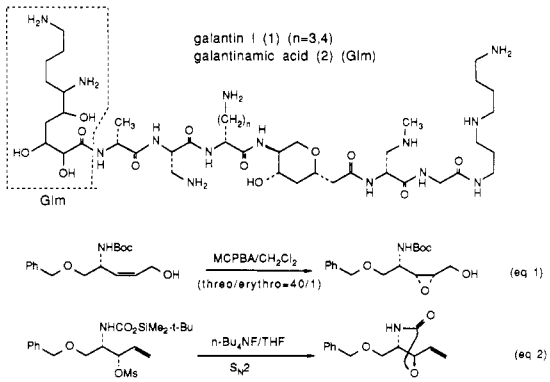
Synthesis and Absolute Structure of Galantinamic Acid

Summary: The structure of galantinamic acid (**2**) has been confirmed to be (2*R*,3*S*,5*S*,6*R*)-**25** by the stereoselective synthesis of each of the eight diastereomers derived from L-lysine; total synthesis of the natural form was accomplished by starting from D-lysine.

Sir: Galantin I (**1**), isolated from a culture broth of *Bacillus pulvifaciens*, has received considerable attention owing to its potent antibacterial activity.[1] The structure of **1**,[2] elucidated by chemical degradation and partly by synthesis, contains a new amino acid named galantinamic acid (**2**)[3] having four chiral centers, of which the stereochemistry remained to be determined. In conjunction with the studies toward the total synthesis of **1**,[2c] we began the structure determination of **2** via the stereoselective syntheses of the eight diastereomers from L- or D-lysine.

Since the primary structure of **2** possesses either a threo or an erythro 1,2-amino hydroxy system[4] at C5 and C6, the stereoselective synthesis of each isomer plays a key role in this study. Initially, we examined an epoxidation of the



(hydroxymethyl)-(*Z*)-allylamine with *m*-chloroperbenzoic acid (MCPBA) and found that the reaction provides the threo epoxide in a highly stereoselective manner (eq 1).[5] On the other hand, the erythro isomer can be prepared, enantiospecifically, from the threo mesylate via fluoride ion treatment of the silyl carbamate derived from *tert*-butyl carbamate (*t*-Boc) (eq 2).[6] On the basis of these methods, the syntheses of the eight diastereomers of **2** starting from L-lysine were carried out as follows.

Synthesis of the Four Diastereomeric Unsaturated Esters. Amino alcohol **3**, prepared from N^{α}-Boc-N^{ϵ}-Z-L-lysine, was converted to the (*Z*)-allyl alcohol **4** [three steps, 65%; (i) SO_3–pyridine, (ii) $(CF_3CH_2O)_2P(O)CH_2CO_2Me$/$KN(SiMe_3)_2$,[7] and (iii) i-$Bu_2AlH/BF_3{\cdot}OEt_2$[8]], which upon treatment with MCPBA gave the threo epoxide **5** (97%; threo/erythro = 40/1) (Scheme I). Regioselective epoxide opening of **5** with $LiAlH_4$ and successive protection of the resulting imino diol **6** furnished silyl ether **9** (three steps, 60%). It was necessary to introduce a *t*-Boc group at the N^{ϵ} position in place of the Z group at this stage because of subsequent chemical transformations and for the final comparison with the natural N^{α},N^{ϵ}-di-Boc derivative. It should be noted that this was carried out in one pot under hydrogenation conditions (H_2/Pd–C, MeOH) in the presence of di-*tert*-butyl dicarbonate (Boc_2O) to yield **10** in 93% yield.[9]

Following the method shown in eq 2, we next examined an inversion of the configuration at C3. Successive treatment of the mesylate **8**, prepared from **7** ($MsCl/Et_3N$), with *tert*-butyldimethylsilyl trifluoromethanesulfonate (TBSOTf)[4a,10] and n-Bu_4NF provided the cyclic carbamate **11** (93%) where the reaction proceeded completely in an S_N2 manner.[6] Conversion of **11** into **12**, an isomeric form of **10**, was carried out by the following sequence of reactions: (i) *dl*-10-camphorsulfonic acid (CSA)/MeOH, (ii) H_2/Pd–C, (iii) $Ba(OH)_2$, (iv) Boc_2O, (v) TBSCl/imidazole, and (vi) $CSA/(CH_3)_2C(OCH_3)_2$; 50% overall yield.[11] Thus, with diastereomeric **10** and **12** in hand, their conversions

(1) Shoji, J.; Sakazaki, R.; Wakishima, Y.; Koizumi, K.; Mayama, M.; Matsuura, S. *J. Antibiot.* **1975**, *28*, 122.

(2) (a) Ando, T.; Terashima, S.; Kawata, M.; Teshima, T.; Wakamiya, T.; Shiba, T. *Peptide Chemistry 1980*; Okawa, K., Ed.; Protein Research Foundation: Osaka, 1981; p 113. (b) Wakamiya, T.; Ando, T.; Teshima, T.; Shiba, T. *Bull. Chem. Soc. Jpn.* **1984**, *57*, 142. (c) Ohfune, Y.; Kurokawa, N. *Tetrahedron Lett.* **1984**, *25*, 1587.

(3) Wakamiya, T.; Terashima, S.; Kawata, M.; Teshima, T.; Shiba, T. *Bull. Chem. Soc. Jpn.* **1988**, *61*, 1422.

(4) For the syntheses of 1,2-amino hydroxyl systems from α-amino acids as the chiral source, see: (a) Sakaitani, M.; Ohfune, Y. *Tetrahedron Lett.* **1987**, *28*, 3987. (b) Reetz, M. T.; Drewes, M. W.; Schmitz, A. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 1141. Other references are cited therein.

(5) (a) Hori, K.; Ohfune, Y. Presented at the 54th Annual Meeting of the Chemical Society of Japan, Tokyo, 1987; Abstract Papers II, p 1033. (b) Similar results were reported independently; see: Kogen, H.; Nishi, T. *J. Chem. Soc., Chem. Commun.* **1987**, 311.

(6) Sakaitani, M.; Hori, K.; Ohfune, Y. *Tennen Yuki Kagobutsu Toronkai Koen Yoshishu* **1986**, *28th*, 526.

(7) For *Z*-selective Horner–Emmons reactions, see: Still, W. C.; Gennari, C. *Tetrahedron Lett.* **1983**, *24*, 4405. A high *Z*/*E* ratio (more than 20/1) when this reaction condition was used was observed in all cases described in the text.

(8) Moriwake, T.; Hamano, S.; Miki, D.; Saito, S.; Torii, S. *Chem. Lett.* **1986**, 815.

(9) For further applications of this method, see: Sakaitani, M.; Hori, K.; Ohfune, Y. *Tetrahedron Lett.*, in press.

(10) Sakaitani, M.; Ohfune, Y. *Tetrahedron Lett.* **1985**, *26*, 5543.

(11) The stereochemistry of **10** and **12** having an isomeric relationship at C3 was confirmed by NOE studies: a NOE (6.8% enhancement) between C3 and C4 protons in the five-membered ring of the erythro isomer **12** was observed, but no NOE was observed in the threo **10**.

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