Stereoselective Hydride Reductions of Cyclic α,*β*-Unsaturated *N*-Diphenylphosphinyl Imines to Protected Allylic Amines

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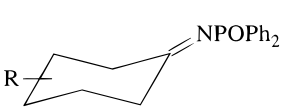
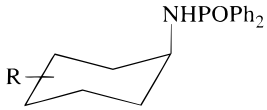
We recently reported the stereoselective hydride reductions of cyclic *N*-diphenylphosphinyl imines to protected primary amines.[1a,b] The reduction of cyclohexyl derivatives **1** with lithium tri-*sec*-butylborohydride was found to provide a highly diastereoselective route to the corresponding axial phosphinylamines **2**, which represent protected versions of primary amines.[1a,b]

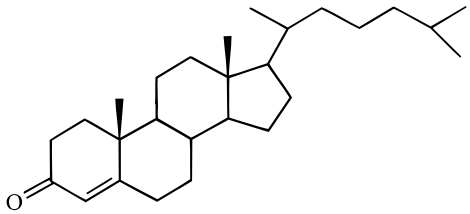


1



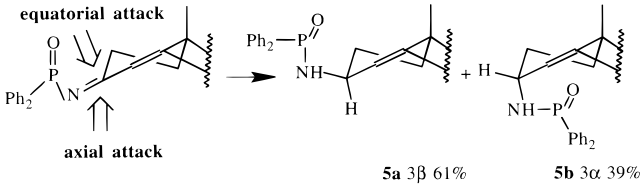
2

However, an attempt to extend this methodology to the reduction of α,*β*-unsaturated cyclohexenyl phosphinyl imines to protected allylic amines[1c] (i.e., **3a**−**c**) led principally to the saturated derivatives **4a**−**c** resulting from reduction of both the phosphinyl imine (to the axial amine derivative) and the *π*-bond (Table 1). Although disappointing, these results were not altogether unexpected because tri-*sec*-butylborohydride has been shown to add preferentially at the 1,4 positions to α,*β*-unsaturated cyclohexenones not further substituted at the *β* position.[2] Also, in line with results obtained from the reduction of *β*-substituted enones,[2,3] reduction of the phosphinyl imine derived from 4-cholesten-3-one (**3d**)



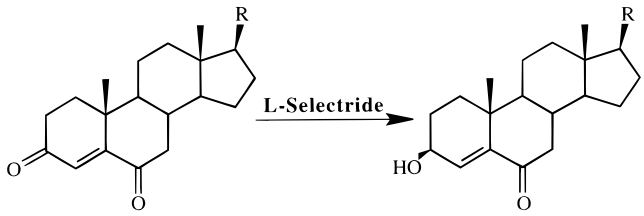
3d

with tri-*sec*-butylborohydride afforded only 1,2-reduction. However, the stereoselectivity obtained was poor, giving a 3:2 mixture of the *β*: α isomers **5d**. Additionally, in contrast to results with cyclohexyl phosphinyl imines,[1a,b] the major product (**5a**) results from an axial approach.



5a 3β 61% **5b** 3α 39%

This reversal of the usual equatorial approach is not unprecedented in that reduction of enone **6** with tri-*sec*-butylborohydride afforded only the *β*-alcohol **7** (arising



6 **7**

from axial attack).[3] In general, axial attack on 2-cyclohexenones is apparently favored relative to that of cyclohexanones,[3,4] and even bulky reagents often prefer axial attack with these types of substrates.[2,4d,e] This has been attributed[5] to better preservation of orbital overlap between the carbon−carbon double bond and the newly forming bond resulting from axial approach. Additionally, theoretical studies on the two possible transition states indicate that the axial attack transition state is almost perfectly staggered, whereas the equatorial approach experiences severe eclipsing.[5] In this particular case, the axially oriented methyl group at C probably supplies additional steric hindrance to equatorial attack. Removal of one of the axial groups (C_3 or C_5 axial hydrogens that create *syn*-1,3-diaxial interactions with reagents approaching axially) that contribute to steric hindrance of axial attack has also been implicated as a factor in the observed increase in attack from this direction.[5] In any case, our objective of obtaining stereoselective reductive processes for amine diastereomers was not met using the nucleophilic tri-*sec*-butylborohydride.

To circumvent the problems associated with nucleophilic conjugate hydride addition to *β*-unsubstituted phosphinyl imines, the utility of the electrophilic reagents 9-borabicyclic [3.3.1]nonane (9-BBN) and diisobutylaluminum hydride (DIBAH) was explored. Indeed, conjugate reduction was suppressed with these reagents, and only 1,2-allylic reduction was obtained with derivatives **3**, as indicated in Table 1. As evident from the table, diastereoselectivity with **3a**−**f** was good to excellent with both reagents affording a predominance of equatorial allylic amine derivatives resulting from axial approach.

The reduction of α,*β*-unsaturated imines to the saturated amine derivatives **4** with tri-*sec*-butylborohydride deserves comment. Although the analogous reduction of

(1) (a) Hutchins, R. O.; Adams, J.; Rutledge, M. *J. Org. Chem.* **1995**, *60*, 7396. (b) Hutchins, R. O.; Zhu, Q.-C.; Adams, J.; Rao, S. J.; Oskay, E.; Abdel-Magid, A. F.; Hutchins, M. G. ACS Symposium Series 641, American Chemical Society: Washington, DC, 1996; 127. (c) For the importance of allylic amines, see: Hutchins, R. O.; Wei, J.; Rao, S. J. *J. Org. Chem.* **1994**, *59*, 4007 and cited references.

(2) Ganem, B. *J. Org. Chem.* **1975**, *40*, 146.

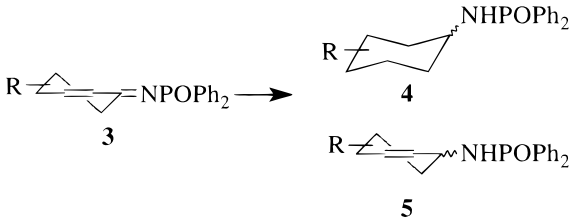
(3) You, Z.; Koreeta, M. *Tetrehedron Lett.* **1993**, *34*, 2745.

(4) (a) Trost, B.; Florez, J.; Jebaratman, D. *J. Am. Chem. Soc.* **1987**, *109*, 613. (b) Kienzle, F.; Mayer, M. *Helv. Chim. Acta* **1978**, *61*, 2609. (c) Stork, G.; Stryker, J. *Tetrahedron Lett.* **1983**, 4887. (d) Idrissi, M.; Santelli, M. *J. Org. Chem.* **1988**, *53*, 1010. (e) Trost, B.; Florez, J.; Haller, K. *J. Org. Chem.* **1988**, *53*, 2396.

(5) Wu, Y.; Houk, K. N.; Florez, J.; Trost, B. *J. Org. Chem.* **1991**, *56*, 3656.

10.1021/jo9812429 CCC: $15.00
Published on Web 10/02/1998

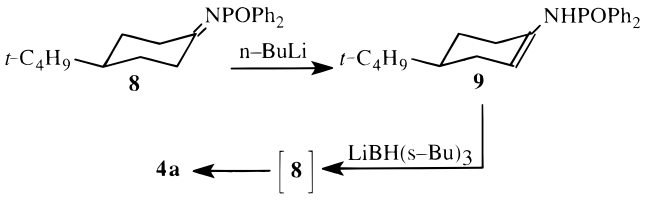
Table 1. Reduction of α,β-Unsaturated Cyclohexylidene *N*-Diphenylphosphinyl Imines



		ratio of axial/equatorial attack on **3**		
			5 (% yield)[a]	
	R	**4** (% yield)[a,b] L-Selectride	9-BBN	DIBAH
3a	4-*tert*-butyl	<5:>95 (42)	89:11 (51)	81:19 (54)
3b	4-*iso*-propyl	<5:>95 (53)	84:16 (54)	78:22 (62)
3c	2-methyl-5-*iso*-propenyl	14:86 (55)	>95:<5 (63)	92:8 (62)
3d	4-cholesten-3-one	(55)[c]	>95:<5 (50)	
3e	3,5-dimethyl	<5:>95 (44)	>95:<5 (59)	>95:<5 (60)
3f	2-isopropylidene-5-methyl		>95:<5 (55)	>95:<5 (53)
3g	4,4-dimethyl		(56)	(53)
3h	3,3,5-trimethyl		(55)	(59)

[a] Yields are for isolated, purified products. Product ratios were determined on the crude products. [b] Characterization of products **4** were provided in ref 1a. [c] Product **5** obtained. Ratio axial:equatorial attack, 61:31.

2-cyclohexenones to the corresponding saturated alcohols with borohydride reagents has been observed,[6] usually a protic solvent is involved which allows the rearrangement of an initially formed enol (from 1,4-reduction to the saturated ketone and subsequent reduction. With tri-*sec*-butylborohydride, cyclic enones in aprotic solvents afford saturated ketones.[2] In the present case, formation of the saturated amine derivatives **4** requires an initial 1,4-reduction of the unsaturated imine **3** followed by reduction of the resulting enamide intermediate. To test this, the phosphinyl enamide **9** was prepared by treatment of the saturated phosphinyl imine **8** (derived from 4-*tert*-butylcyclohexanone oxime[1a]) with *n*-butyllithium. Subsequent reduction with tri-*sec*-butylborohydride afforded the saturated derivative **4a** (*cis* diastereomer). The



diastereoselectivity is consistent with previous results obtained in the reduction of **8** with the same reagent.[1a] Repetition of the reduction of **9** with triethylborodeuteride resulted in deuterium incorporation at C_1 in **4a**, indicating rearrangement of the phosphinyl enamide intermediate prior to deuteride (or hydride) delivery. The details of this unusual rearrangement in the absence of a protic solvent are under current investigation.

In summary, the reduction of α,β-unsaturated cyclohexenyl *N*-diphenylphosphinyl imines with the electrophilic hydride reagents DIBAH and 9-BBN affords protected versions of primary 2-cyclohexenyl allylic amines (primarily from axial attack), whereas trialkylborohydrides give the corresponding saturated derivatives resulting from conjugate addition of hydride, rearrangement, and subsequent imine reduction resulting from equatorial attack.

(6) (a) Brown, H. C.; Hess, H. M. *J. Org. Chem.* **1969**, *34*, 2206. (b) Dilling, W. L.; Piepys, R. A. *J. Org. Chem.* **1970**, *35*, 2971. (c) Johnson, M. R.; Rickborn, B. *J. Org. Chem.* **1970**, *35*, 1041. (d) Varma, R. S.; Kabalka, G. W. *Synth. Commun.* **1985**, *15*, 985. (e) Elliott, J.; Hall, D.; Warren, S. *Tetrahedron Lett.* **1989**, *30*, 601.

Experimental Section

General Information. ^{1}H nuclear magnetic resonance spectra were recorded at 250 MHz with $CDCl_3$ as solvent and tetramethylsilane as an internal reference. ^{31}P nuclear magnetic resonance spectra were recorded at 36.23 MHz with $CDCl_3$ as a solvent and phosphoric acid (H_3PO_4) as an external reference (0 ppm). Elemental analyses were performed by the University of Pennsylvania. Melting points are uncorrected.

Materials. All solvents were freshly distilled under an atmosphere of argon prior to use. THF was distilled from sodium/benzophenone ketyl. Methylene chloride was distilled from P_2O_5. Petroleum ether and triethylamine were distilled from calcium hydride. Chlorodiphenylphosphine was fractionally distilled under reduced pressure and stored under argon. All unsaturated ketones were purchased from Aldrich except 4-*tert*-butyl-2-cyclohexenone which was prepared via a literature procedure.[7] All organic solutions were dried with magnesium sulfate unless otherwise specified.

General Procedure for the Preparation of Oximes. A 250-mL round-bottom flask equipped with a reflux condenser was charged with hydroxylamine hydrochloride (40 mmol, 2.78 g), sodium acetate trihydrate (40 mmol, 5.44 g), and water (50 mL). The resulting solution was heated to ca. 60 °C, and the appropriate ketone (20 mmol) in methanol was added. Enough methanol was then added to give a clear solution, and stirring at 60 °C was continued overnight. The resulting solution was cooled to room temperature, diluted with water (100 mL), and extracted with ether (3 × 25 mL). The combined organic phase was washed with saturated sodium bicarbonate (2 × 10 mL) and brine and then dried. The solvent was removed at reduced pressure, and the crude oximes were purified either by recrystallization or by column chromatography.

4-*tert*-Butyl-2-cyclohexenone oxime: yield, 75%; mp 76−77 °C; ^{1}H NMR ($CDCl_3$) 10.2−10.4 (br, 1H, 6.0−6.2 (q, 2H), 3.1−3.2 (m, 1H), 1.9−2.0 (m, 2H), 1.3−1.4 (m, 2H), 0.9 (s, 9H). ^{13}C NMR ($CDCl_3$) 158.0, 141.5, 139.0, 124.6, 112.6, 48.6, 46.6, 33.0, 27.8, 22.6. Anal. Calcd for $C_{10}H_{17}NO$: C, 71.80; H, 10.17: N, 8.38. Found: C, 71.51; H, 8.53; N, 8.26.

4-Isopropyl-2-cyclohexenone oxime: yield, 84%; mp 70−71 °C (lit.[8a] mp 69−70.5 °C). Anal. Calcd for $C_9H_{15}NO$: C, 70.55; H, 9.87; N, 9.14. Found: C, 70.83; H, 10.19; N, 9.25.

(7) Garbisch, E. *J. Org. Chem* **1965**, *30*, 2109.

2-Methyl-5-isopropenyl-2-cyclohexenone (carvone) oxime: yield, 89%; mp 70−71 °C (lit.[8b] mp 73.1−73.5 °C).

4-Cholesten-3-one oxime: yield, 90%; mp 145−148 °C (1:3 *E*:*Z* mixture) [lit.[8c] mp 152−153 °C (2:3 *E*:*Z* mixture)].

3,5-Dimethyl-2-cyclohexenone oxime: yield, 92%; mp 69−70 °C (lit.[8d] mp 70.5−73.5 °C).

2-Isopropylidene-5-methylcyclohexanone (pulegone) oxime: yield, 78%; viscous oil. Anal. Calcd for $C_{10}H_{17}NO$: C, 71.85; H, 10.17; N, 8.38. Found: C, 71.47; H, 9.91; N, 8.05.

4,4-Dimethyl-2-cyclohexenone oxime: yield, 90%; mp 48−48.5 °C (lit.[8e] "low melting").

3,5,5-Trimethyl-2-cyclohexenone oxime: yield: 93%; mp 73−74 °C (lit.[8b] mp 73.1−73.7 °C).

General Procedure for the Synthesis of *N*-Diphenylphosphinyl Imines. The appropriate oxime (2.4 mmol) was added to an oven-dried 100-mL three-neck flask kept under argon. Dry methylene chloride (25 mL), dry petroleum ether (25 mL), and triethylamine (2.4 mmol) were added, and the resulting solution was cooled to −40 °C. To this solution was added chlorodiphenylphosphine (2.4 mmol) via syringe, and the mixture was allowed to stir at −40 °C for 1.5−2 h (white precipitate of triethylamine hydrochloride was observed). The mixture was then cooled to −78 °C and transferred via cannulla (positive argon or nitrogen pressure) to a filter under the inert atmosphere. The resulting clear colorless solution was carefully evaporated (high vacuum bled through a $CaSO_4$ drying tube) at 0 °C. The crude phosphinyl imines were immediately subjected to the appropriate reducing agent as described below.

General Procedure for the Reduction of *N*-Diphenylphosphinyl Imines with Lithium Tri-*sec*-butylborohydride (L-Selectride). To a solution of crude phosphinyl imine (1.5 mmol) in dry tetrahydrofuran (20 mL) was added L-Selectride (3.2 mmol, 3.2 mL of 1.0 M solution in THF). The resulting yellow solution was stirred for 3 h at room temperature under argon and then quenched with water (2 mL). The organoboranes were decomposed via one of two methods: In method A, the solution was cooled to 0 °C and 15% sodium hydroxide (0.5 mL) was added followed by the slow addition of 30% hydrogen peroxide (0.5 mL). The cooling bath was removed, and the solution was stirred for 30 min. Water (15 mL) and ether (25 mL) were then added. The layers were separated, and the aqueous layer was extracted with ether (2 × 15 mL). The combined organics were washed with 30% sodium bisulfite (2 × 20 mL) and brine and then dried. In method B, sodium perborate tetrahydrate, $NaBO_3 \cdot 4H_2O$, (1.48 g, 9.6 mmol) was added, and the mixture was stirred at room temperature for 2 h. Water (15 mL) and ether (30 mL) were added and the layers were separated. The aqueous layer was extracted with ether (2 × 25 mL). The combined organic phase was washed with brine and dried. Evaporation and flash column chromatography yielded the products.

General Procedure for the Reduction of *N*-Diphenylphosphinyl Imines with Diisobutyl Aluminum Hydride (DIBAH). To the appropriate phosphinyl imine (1.8 mmol) under argon in dry tetrahydrofuran (20 mL) at room temperature was added DIBAH (3.9 mmol, 1.0 M in THF). The resulting solution was stirred overnight at room temperature. The reaction was quenched with water (2 mL) and 15% sodium hydroxide (2 mL) and allowed to stir for 30 min. Water (30 mL) was then added, and the solution was extracted with ether (3 × 25 mL). The combined organic solutions were washed with water and brine and dried. Evaporation and flash chromatography (1:1 hexanes/ethyl acetate) afforded the products.

General Procedure for the Reduction of *N*-Diphenylphosphinyl Imines with 9-Borobicyclo[3.3.1]nonane (9-BBN). To the appropriate phosphinyl imine (1.8 mmol) under argon in dry tetrahydrofuran (20 mL) at room temperature was added 9-BBN (4.0 mmol, 0.5 M in THF). The resulting solution was stirred for 40 h at room temperature. The reaction was quenched with water (2 mL) and allowed to stir for 30 min. Water (25 mL) was then added, and the solution was extracted with ether (3 × 25 mL). The combined organic solutions were washed with water and brine and dried. Evaporation and flash chromatography (1:1 hexanes/ethyl acetate) afforded the products.

Diastereoselective Reduction Results of Cyclic *N*-Diphenylphosphinyl Cyclohexenyl Imines. Experimental procedures are as given above. Ratios of diastereomeric products were determined by ^{31}P NMR measurements on the crude product mixtures. Identification and characterization of the saturated cyclohexyl *N*-diphenylphosphinyl amine products (**4a**−**c,d**) was accomplished by comparison with authentic samples.[1a] Characterization of the major cyclohexenyl phosphinylamine diastereomers of **5** are presented below.

***trans*-*N*-(Diphenylphosphinyl)-4-*tert*-butyl-2-cyclohexenylamine (5a):** mp 144−146 °C (from 9:1 hexane/ethyl acetate); ^{31}P NMR ($CDCl_3$) 23.75. Anal. Calcd for $C_{22}H_{28}NOP$: C, 74.78; H, 7.93; N, 3.96. Found: C, 74.49; H, 7.28; N, 3.83.

***trans*-*N*-(Diphenylphosphinyl)-4-isopropyl-2-cyclohexenylamine (5b):** mp 148−149 °C (from 9:1 hexane/ethyl acetate); ^{31}P NMR ($CDCl_3$) 23.95. Anal. Calcd for $C_{21}H_{26}NOP$: C, 74.31; H, 7.72. Found: C, 73.97; H, 7.71.

***cis*-*N*-(Diphenylphosphinyl)-2-methyl-5-isopropenyl-2-cyclohexenylamine (5c):** mp 143−145 °C (from 9:1 hexane/ethyl acetate); ^{31}P NMR ($CDCl_3$) 22.97. Anal. Calcd for $C_{22}H_{26}NOP$: C, 75.21; H, 7.40; N, 3.98. Found: C, 75.31; H, 8.20; N, 3.64.

***N*-(Diphenylphosphinyl)-4-cholesten-3*β*-amine (5d):** mp 217 °C (dec, from 9:1 hexane/ethyl acetate); ^{13}P NMR ($CDCL_3$) 23.56. Anal. Calcd for $C_{39}H_{56}NOP$: C, 79.96; H, 9.63; N, 2.39. Found: C, 79.71; H, 9.80; N, 2.16.

***cis*-*N*-(Diphenylphosphinyl)-3,5-dimethyl-2-cyclohexenylamine (5e):** mp 140−142 °C (from 9:1 hexane/ethyl acetate); ^{31}P NMR ($CDCl_3$) 23.88. Anal. Calcd for $C_{20}H_{24}NOP$: C, 73.84; H, 7.38; N, 4.30. Found: C, 73.65; H, 7.46; N, 3.59.

***cis*-*N*-(Diphenylphosphinyl)-2-isopropenyl-5-methyl-2-cyclohexenylamine (5f):** mp 108−110 °C (from 9:1 hexane/ethyl acetate); ^{31}P NMR ($CDCl_3$) 22.68. Anal. Calcd for $C_{22}H_{28}NOP$: C, 73.94; H, 7.84; N, 3.92. Found: C, 73.81; H, 7.70; N, 3.82.

***N*-(Diphenylphosphinyl)-4,4-dimethyl-2-cyclohexenylamine (5 g):** mp 142−144 °C (from 9:1 hexane/ethyl acetate); ^{31}P NMR ($CDCl_3$) 23.42. Anal. Calcd for $C_{20}H_{24}NOP$: C, 73.84; H, 7.38; N, 4.30. Found: C, 73.67; H, 7.71; N, 3.62.

***N*-(Diphenylphosphinyl)-3,3,5-trimethyl-2-cyclohexenylamine (5h):** mp 140−142 °C (from 9:1 hexane/ethyl acetate); ^{31}P NMR ($CDCl_3$) 23.75. Anal. Calcd for $C_{21}H_{26}NOP$: C, 74.31; H, 7.72; Found: C, 73.94; H, 7.84.

***N*-(Diphenylphosphinyl)-*N*-(4-*tert*-butylcyclohex-1-ene)-amine (9).** To a solution of 4-*tert*-butylcyclohexyl oxime[1a] (0.80 g, 4.7 mmol) and triethylamine (0.72 mL, 5.2 mmol) in 1:1 methylene chloride/hexanes (mL) at −40 °C was added chlorodiphenylphosphine (0.95 mL, 5.2 mmol) via syringe. The temperature immediately rose to −30 °C. After addition was complete, the reaction mixture was stirred at −40 °C for 2 h and then filtered under nitrogen to remove triethylamine hydrochloride. The solvent was removed under vacuum, dry THF (30 mL) was added, and the solution of **8** was cooled to −40 °C. To this was added *n*-BuLi (1 M, 5.2 mL, 5.2 mmol) dropwise, and the solution was stirred for 2 h. The reaction was then quenched with water at 0 °C and extracted with ether. The organic phase was washed with brine and dried over anhydrous Na_2SO_4. Evaporation and recrystallization from 9:1 hexanes/ethyl acetate afforded **9** as white crystals: mp 134−135 °C; ^1H NMR ($CDCl_3$) 7.3−7.9 (m, 10H), 5.0−5.2 (br s, 1H), 4.3−4.5 (d, 1H), 2.0−2.2 (m, 2H), 1.6−1.9 (m, 2H), 1.6−1.9 (m, 3H), 0.8 (s, 9H); ^{31}P NMR ($CDCl_3$) 18.27; ^{13}C NMR ($CDCl_3$) 131.7, 131.6, 131.5, 128.4, 128.2, 107.3, 43.4, 31.9, 30.5, 27.0, 25.2, 23.8. Anal. Calcd for $C_{22}H_{28}NOP$: C, 74.78; H, 7.93; N, 3.96. Found: 74.49; H, 7.28; N, 3.86.

***cis*-*N*-(Diphenylphosphinyl)-4-*tert*-butycyclohexylamine (4a).** To a solution of **9** (0.40 g, 1.1 mmol) in dry THF was added L-Selectride (1 M, 2.8 mL, 2.2 mmol) dropwise at room temperature. The reaction mixture was stirred for 2.5 h, quenched with 3 equiv of sodium perborate, and extracted with ether. The organic phase was dried (Na_2SO_4), and the solvent was removed at reduced pressure. The crude product obtained

(8) (a) Adams, J. Ph.D. Thesis, Drexel University, 1994. (b) Montgomery, R. S.; Doughery, C. *J. Org. Chem.* **1952**, *17*, 823. (c) Suginome, H.; Kaji, M.; Yamada, S. *J. Chem. Soc., Perkin Trans. 1* **1988**, 321. (d) Horning, E. C.; Stromberg, V. I.; Llyod, H. A. *J. Am. Chem. Soc.* **1952**, *74*, 5153. (e) Wolkowski, Z. W.; Thoai, N.; Weimann, J. *Tetrahedron Lett.* **1970**, 93.

was purified by flash chromatography (7:3 hexanes/ethyl acetate) to obtain **4a** (0.375 g, 94%), which was identical in all respects with an authentic sample.[1a]

Supporting Information Available: 250 MHz ^{1}H and ^{13}C NMR of the *N*-diphenylphosphinyl-cyclohexenylamines **5a**−**c,e**−**h** (2 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

JO9812429

Additions and Corrections

Vol. 62, 1997

Tadashi Eguchi, Kenji Arakawa, Takumi Terachi, and Katsumi Kakinuma*. Total Synthesis of Archaeal 36-Membered Macrocyclic Diether Lipid.

Page 1924, Scheme 5. The preparation of phytanol by a similar approach of an asymmetric hydrogenation of phytol using a different Ru(II) catalyst had been reported earlier by Prof. L. R. Sita, and was inadvertently omitted from our citation. The reference is as follows (we thank Prof. L. R. Sita for this information): Sita, L. R. *J. Org. Chem.* **1993**, *58*, 5285.

JO9840168

10.1021/jo9840168
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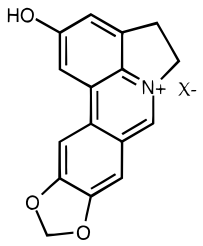
Aaron E. Bunnell, Lee A. Flippin,* and Yanzhou Liu. Total Synthesis of (±)-Roserine.

Page 9305. The correct structure of ungeremine is



ungeremine

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Vol. 63, 1998

Xavier Creary* and Jennifer Tricker. Reaction of Benzylic α-Hydroxythioamides with Thionyl Chloride.

Page 4910. ^{1}H NMR spectral data for the key compounds **2** and **6** should be exchanged in the Experimental Section. The corrected data should read: ^{1}H NMR of **2** ($CDCl_3$) δ 7.723 (d of m, J = 7.5 Hz, 1 H), 7.584 (d of m, J = 7.5 Hz, 1 H), 7.450 (t of d, J = 7.5, 1.3 Hz, 1 H), 7.366 (t of d, J = 7.5, 1.3 Hz, 1 H), 3.48 (br, 3 H), 2.36 (br, 3 H). ^{1}H NMR of **6** ($CDCl_3$) δ 7.732 (d of m, J = 7.3 Hz, 1 H), 7.562 (d of m, J = 7.3 Hz, 1 H), 7.455 (t of d, J = 7.3, 1.3 Hz, 1 H), 7.374 (t of d, J = 7.3, 1.3 Hz, 1 H), 2.87 (br, 3 H), 2.01 (br, 3 H). ^{13}C NMR data are correct as reported.

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Published on Web 10/14/1998