ORGANIC LETTERS

2005
Vol. 7, No. 4
553−556

Samarium Diiodide Promoted Generation and Asymmetric Hydroxyalkylation of *N,O*-Diprotected (3*S*)-3-Pyrrolidinol 2-Carbanions

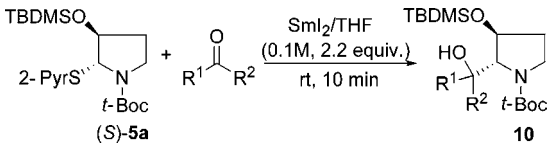
Xiao Zheng, Chen-Guo Feng, Jian-Liang Ye, and Pei-Qiang Huang*

Department of Chemistry and The Key Laboratory for Chemical Biology of Fujian Province, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen, Fujian 361005, P. R. China

pqhuang@xmu.edu.cn

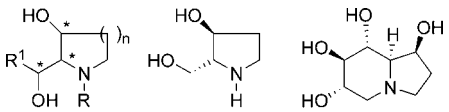
Received November 4, 2004

ABSTRACT



The *N,O*-diprotected chiral nonracemic 2-pyridyl 3-pyrrolidinol-2-yl sulfide 5a undergoes efficient SmI_2 mediated reduction to give the *N,O*-diprotected 3-pyrrolidinol 2-carbanion intermediate D, which reacted under Barbier-type conditions with ketones and aldehydes to afford the protected *N*-α-hydroxyalkyl-3-pyrrolidines 10b–h with excellent diastereoselectivity at the newly formed chiral center in the pyrrolidine ring. Application of the present method led to the formal asymmetric syntheses of (2*R*,3*S*)-2-hydroxymethyl-3-pyrrolidinol (2) and (2*S*,3*S*)-3-hydroxyproline (12).

2-Hydroxyalkyl-3-pyrrolidinols **1a** and their higher homologues **1b** are key structural features found in a number of polyhydroxylated bioactive natural products, which include pyrrolidine, pyrrolizidine, and indolizidine alkaloids.[1] For example, (2*R*,3*S*)-2-hydroxymethyl-3-pyrrolidinol (**2**) (CYB-3)[2] and castanospermine (**3**) are alkaloids isolated from the seeds of *Castanospermum australe*.[3a] Castanospermine has also been isolated from the dried pod of *Alexa leiopetala*,[3b] and was found to be a powerful inhibitor of α- and β-glucosidases.[4] The important bioactivities exhibited by these alkaloids and/or azasugars have stimulated considerable synthetic interest and a number of strategies have been developed for the syntheses of these natural products.[1] However, efficient and straightforward approaches continue to be highly desirable.



1 (a. n=1; b. n=2) (2*R*,3*S*)-**2** **3**

A conceptually attractive approach to 2-hydroxyalkyl-3-pyrrolidinols involves generation of the chiral nonracemic

(1) For recent reviews of synthesis and biological activity, see: (a) Nemr, A. E. *Tetrahedron* **2000**, *56*, 8579. (b) Asano, N.; Nash, R. J.; Molyneux, R. J.; Fleet, G. W. J. *Tetrahedron*: *Asymmetry* **2000**, *11*, 1645. (c) Sears, P.; Wong, C.-H. *Angew. Chem., Int. Ed.* **1999**, *38*, 2300. (d) Michael, J. P. *Nat. Prod. Rep.* **1999**, *16*, 675. (e) Elbein, A. D.; Molyneux, R. J. In *Iminosugars as Glycosidase Inhibitors*; Stutz, A. E., Ed.; Wiley-VCH: Weinheim, Germany, 1999; p 216. (f) Michael, J. P. *Nat. Prod. Rep.* **1997**, *14*, 619. (g) Takahata, H.; Momose, T. In *The Alkaloids*; Cordell, G. A., Ed.; Academic: San Diego, CA, 1993; Vol. 44, Chapter 3. (h) Elbein, A. D.; Molyneux, R. In *Alkaloids*: *Chemical and Biological Perspectives*; Pelletier, S. W., Ed.; Wiley and Sons: New York, 1987; Vol. 5.

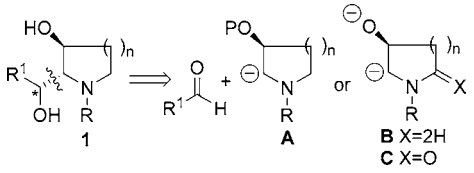
(2) Nash, R. J.; Bell, E. A.; Fleet, G. W. J.; Jones, R. H.; Williams, J. M. *J. Chem. Soc., Chem. Commun.* **1985**, 738.

(3) (a) Hohenschutz, L. D.; Bell, E. A.; Jewess, P. J.; Leworthy, D. P.; Pryce, R. J.; Arnold, E.; Clardy, J. *Phytochemistry* **1981**, *20*, 811. (b) Nash, R. J.; Fellows, L. E.; Dring, J. V.; Stirton, C. H.; Carter, D.; Hegarty, M. P.; Bell, E. A. *Phytochemistry* **1988**, *27*, 1403.

(4) (a) Campbell, B. C.; Molyneux, R. J.; Jones, K. C. *J. Chem. Ecol.* **1987**, *13*, 1759. (b) Pan, Y. T.; Hori, H.; Saul, R.; Sanford, B. A.; Molyneux, R. J.; Elbein, A. D. *Biochemistry* **1983**, *22*, 3975. (c) Saul, R.; Chambers, J. P.; Molyneux, R. J.; Elbein, A. D. *Arch. Biochem. Biophys.* **1983**, *221*, 593. (d) Szumilo, T.; Kaushal, G. P.; Elbein, A. D. *Arch. Biochem. Biophys.* **1986**, *247*, 261.

10.1021/ol047733z CCC: $30.25 © 2005 American Chemical Society
Published on Web 01/21/2005

Scheme 1. Retrosynthetic Analysis of 2-Hydroxyalkyl 3-Pyrrolidinols **1a** and Their Higher Homologues **1b**



3-pyrrolidinol 2-carbanion synthon **A** (Scheme 1). However, this remains a challenging problem in carbanion chemistry despite much effort having been paid, and a number of umpoled methods having been developed, for the generation of *N*-α-carbanions in general,[5] in particular 2-lithiopyrrolidines or 2-lithiopiperidines.[6,7] Past attempts at generating the α-carbanion synthons **A**, **B**, or **C** suffered from *β*-elimination (for **A**, P = Me),[8] or wrong regioselectivity (for **B**),[9] or quick proton exchange (for **C**).[10] Only in a specific case, where a coordinating group (methoxycarbonyl) was present in the C-2 position, was the 3-hydroxylate 2-lithiopyrrolidine generated.[11] Alternatively, both achiral[12] and chiral nonracemic[13] synthetic equivalents of **A** have been reported. In continuation of our interests in developing substituted 2-lithiopyrrolidine-based synthetic methodologies for alkaloid synthesis,[10,13] we wish to report herein our results on the development of a samarium diiodide (SmI_2) mediated α-hydroxyalkylation method[7] using *N*,*O*-diprotected 2-pyrrolidinyl sulfides **5** as the synthetic equivalents of the chiral nonracemic 3-pyrrolidinol 2-carbanion synthons **A**.

Inspired by the SmI_2-mediated[14] C-glycosylation chemistry developed by Beau[15] and Skrydstrup,[16] the sulfones **4** and sulfides **5** were designed as the synthetic equivalents of **A** (P = TBDMS, Bn) (Figure 1).

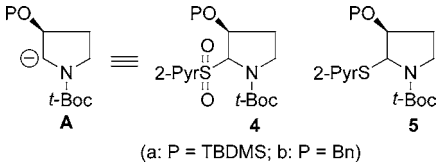
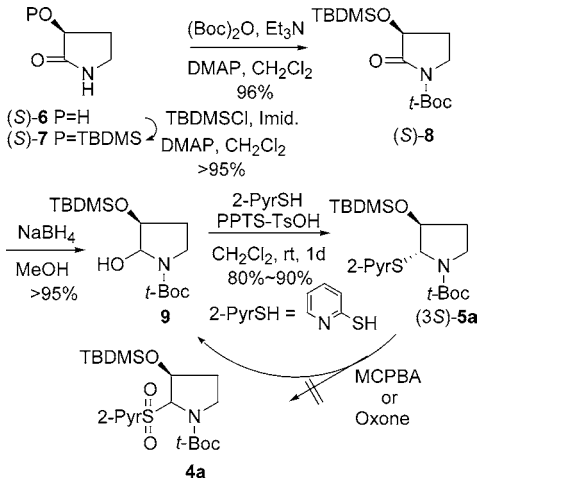


Figure 1. Synthetic equivalents of synthons **A**.

We first selected sulfone **4a** (P = TBDMS) as a precursor of synthon **A**. However, the failure to prepare **4a**[17] led us to give up sulfones **4** (P = TBDMS, Bn) and turn to sulfides **5** (P = TBDMS, Bn) as the synthetic equivalents of **A**. The synthesis of **5a** is depicted in Scheme 2. Known (*S*)-3-hydroxy-2-pyrrolidinone[18] (**6**) was silylated (TBDMSCl, imidazole, DMAP, CH_2Cl_2, room temperature, 24 h) to give (*S*)-**7** in 95% yield. Treatment of (*S*)-**7** with di-*tert*-butyl dicarbonate in the presence of both triethylamine and a

Scheme 2. Synthesis of *N*,*O*-Diprotected 2-Pyrrolidinyl Sulfide **5a**



(5) For reviews involving the generation and application of α-lithioamines, see: (a) Cohen, T.; Bhupathy, M. *Acc. Chem. Res.* **1989**, *22*, 152. (b) Gant, T. G.; Meyers, A. I. *Tetrahedron* **1994**, *50*, 2297. (c) Beak, P.; Basu, A.; Gallagher, D. J.; Park, Y. S.; Thayumanavan, S. *Acc. Chem. Res.* **1996**, *29*, 552. (d) Yus, M. *Chem. Soc. Rev.* **1996**, *25*, 155. (e) Cohen, T. *Pure Appl. Chem.* **1996**, *68*, 913. (f) Gawley, R. E. *Curr. Org. Chem.* **1997**, *1*, 71. (g) Kessar, S. V.; Singh, P. *Chem. Rev.* **1997**, *97*, 721. (h) Katritzky, A.; Qi, M. *Tetrahedron* **1998**, *54*, 2647. (i) Husson, H. P.; Royer, J. *Chem. Soc. Rev.* **1999**, *28*, 383. (j) Rassu, G.; Zanardi, F.; Battistini, L.; Casiraghi, G. *Chem. Soc. Rev.* **2000**, *29*, 109.

(6) For representative approaches to optically active α-lithiopyrrolidines and α-lithiopiperidines, see: (a) Meyers, A. I.; Dickman, D. A.; Bailey, T. R. *J. Am. Chem. Soc.* **1985**, *107*, 7974. (b) Huang, P.-Q.; Arseniyadis, S.; Husson, H.-P. *Tetrahedron Lett.* **1987**, *28*, 547. (c) Gawley, R. E.; Hart, G. C.; Bartolotti, L. J. *J. Org. Chem.* **1989**, *54*, 175. (d) Pearson, W. H.; Lindbeck, A. C.; Kampf, J. W. *J. Am. Chem. Soc.* **1993**, *115*, 2622. (e) Wu, S.; Lee, S.; Beak, P. *J. Am. Chem. Soc.* **1996**, *118*, 715. (f) Coldham, I.; Hufton, R.; Snowden, D. *J. Am. Chem. Soc.* **1996**, *118*, 5322. (g) Wiberg, K. B.; Bailey, W. F. *Angew. Chem., Int. Ed.* **2000**, *39*, 2127. (h) Dearden, M. J.; Firkin, C. R.; Hermet, J.-P. R.; O'Brien, P. *J. Am. Chem. Soc.* **2002**, *124*, 11870. (i) Watson, R. T.; Gore, V. K.; Chandupatla, K. R.; Dieter, R. K.; Snyder, J. P. *J. Org. Chem.* **2004**, *69*, 6105.

(7) For samarium diiodide mediated generation of pyrrolidine and piperidine α-carbanions, see: (a) Collin, J.; Namy, J. L.; Kagan, H. B. *Tetrahedron Lett.* **1992**, *33*, 2973. (b) Murakami, M.; Hayashi, M.; Ito, Y. *J. Org. Chem.* **1992**, *57*, 794. (c) Booth, S. E.; Benneche, T.; Undheim, K. *Tetrahedron* **1995**, *51*, 3665.

(8) Beak, P.; Lee, W. K. *J. Org. Chem.* **1993**, *58*, 1109.

(9) (a) Sunose, M.; Peakman, T. M.; Charmant, J. P. H.; Gallagher, T.; Macdonald, S. J. F. *Chem. Commun.* **1998**, 1723. (b) Pandey, G.; Chakrabarti, D. *Tetrahedron Lett.* **1998**, *39*, 8371 and references therein.

(10) Huang, P.-Q.; Zheng, X.; Wang, S.-L.; Ye, J.-L.; Jin, L.-R.; Chen, Z. *Tetrahedron*: *Asymmetry* **1999**, *10*, 3309.

(11) (a) Williams, R. M.; Cao, J. *Tetrahedron Lett.* **1996,** *37*, 5441. (b) Williams, R. M.; Cao, J.; Tsujishima, H. *Angew. Chem., Int. Ed.* **2000**, *39*, 2540.

(12) (a) Gallagher, T.; Giles, M.; Subramanian, R. S.; Hadley, M. S. *J. Chem. Soc., Chem. Commun.* **1992**, 166. (b) S. H. J.; Subramanian, R. S.; Roberts, J. K.; Hadley, M. S. *J. Chem. Soc., Chem. Commun.* **1994**, 933.

(13) (a) Huang, P.-Q.; Wu, T.-J.; Ruan, Y.-P. *Org. Lett.* **2003**, *5*, 4341. (b) Huang, P.-Q.; Deng, J. *Synlett* **2004**, 247. (c) Tang, T.; Ruan, Y.-P.; Ye; J.-L.; Huang, P.-Q. *Synlett* **2005**, 231.

(14) For an excellent review, see: Kagan, H. B. *Tetrahedron* **2003**, *59*, 10351.

(15) (a) Miquel, N.; Doisneau, G.; Beau, J. M. *Angew. Chem., Int. Ed.* **2000**, *39*, 4111. (b) Skrydstrup, T.; Jarreton, O.; Mazeas, D.; Urban, D.; Beau, J. M. *Chem. Eur. J.* **1998**, *4*, 655. (c) Urban, D.; Skrydstrup, T.; Beau, J. M. *J. Org. Chem.* **1998**, *63*, 2507.

(16) Mikkelsen, L. M.; Krintel, S. L.; Jimenez-Barbero, J.; Skrydstrup, T. *Chem. Commun.* **2000**, 2319.

(17) Attempts to oxidize **5a** to sulfone **4a** with either *m*-CPBA or oxone failed. Instead, the hydrolyzed product was always obtained.

(18) Huang, P.-Q.; Zheng, X.; Wei, H. *Heterocycles* **2003**, *60*, 1833.

catalytic amount of DMAP provided (*S*)-**8** in 96% yield. Controlled regioselective reduction[19] of (*S*)-**8** with $NaBH_4$ at 0 °C yielded a diastereomeric mixture of *N*,*O*-hemiacetal **9** in 95% yield. TLC monitoring of the reaction showed that **9** was formed as only one diastereomer, which partially and readily epimerized during the workup procedure and also upon standing. Reaction of **9** with 2-mercaptopyridine in the presence of 1% (w/w) of a 1:1 mixture of PPTS and TsOH yielded the desired **5a** in nearly diastereomerically pure form, which epimerized and hydrolyzed readily.

With multigram quantities of **5a** available, SmI_2-mediated hydroxyalkylations were investigated. To test the possibility of using **5a** as a synthetic equivalent of **A**, a THF solution of **5a** was treated, at 20 °C and in the presence of an excess of MeOH, with 2.2 molar equiv of SmI_2 (0.1 M in THF) for 10 min. In such a way, the desired protected (*S*)-3-pyrrolidinol (**10a**) was isolated in 95% yield (Scheme 3,

Scheme 3. SmI_2-Mediated Hydroxyalkylations of **5a** and **5b**

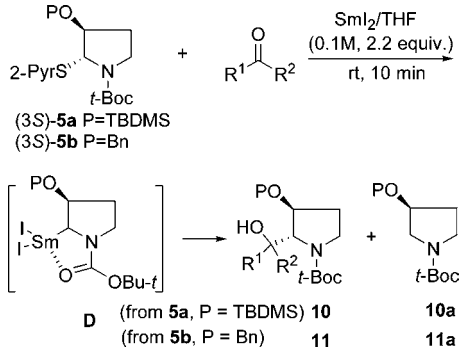


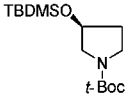
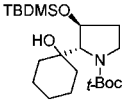
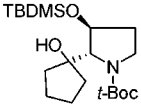
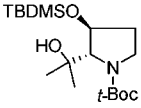
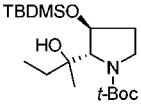
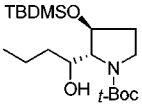
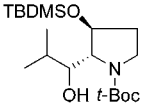
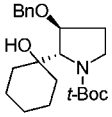
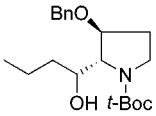
Table 1, entry 1). The formation of **10a** in high yield implied that the reductive samariation of **5a** occurred smoothly, and the presumed organosamarium (III) intermediate **D** was stable to β-elimination.

Encouraged by this result, we then proceeded to study the reductive hydroxyalkylation of **5a** under Barbier-type conditions. Happily, treatment of a mixture of cyclohexanone (1.5 molar equiv) and **5a** in THF with freshly prepared SmI_2 (0.1 M in THF, 2.2 molar equiv) at room temperature, followed by stirring at room temperature for 10 min (protocol A), afforded the desired reductive coupling product **10b** in 50% yield, alongside the protonated product **10a** in 33% yield (Table 1, entry 2). Better yields (62%) of **10b** could be obtained by using an alternative procedure (protocol B), which consisted of adding freshly prepared SmI_2 (0.1 M in THF, 4.0 molar equiv) to a mixture of cyclohexanone (2.0 molar equiv) and **5a** in THF at 0 °C, followed by warming the reaction mixture to room temperature over 10 min (Table 1, entry 3).

The desired product **10b** was formed exclusively according to TLC analysis and chromatographic separation of the crude reaction mixture. The homogeneity of **10b** was confirmed

(19) Wunberg, J. B. P. A.; Schoemaker, H. E.; Speckamp, W. N. *Tetrahedron* **1978**, *34*, 179.

Table 1. The Results for the Reductive Coupling of **5a**/**5b** with Electrophiles (Scheme 3)[20]

Entry	Sulfide (5a/5b) / El	Product (% Yield)[a] (d.r.)[b]	Side product (% Yield)[a]
1	**5a** / MeOH	 **10a** (95)	–
2	**5a** / $(CH_2)_5CO$	 **10b** (50)[c]	**10a** (33)
3	**5a** / $(CH_2)_5CO$	**10b** (62)[d]	**10a** (33)
4	**5a** / $(CH_2)_4CO$	 **10c** (58)[d]	**10a** (32)
5	**5a** / MeCOMe	 **10d** (81)[d]	**10a** (8)
6	**5a** / MeCOEt	 **10e** (70) (56:44)[c]	**10a** (17)
7	**5a** / MeCOEt	**10e** (73)(55:45)[d]	**10a** (21)
8	**5a** / *n*-PrCHO	 **10f** (92) (42:58)[c]	**10a** (5)
9	**5a** / *i*-PrCHO	 **10g** (60) (39:61)[c]	**10a** (32)
10	**5b** / $(CH_2)_5CO$	 **11b** (52)[d]	**11a** (35)
11	**5b** / *n*-PrCHO	 **11c** (63) (45:55)[c]	**11a** (32)

[a] Isolated yield. [b] Diastereoselectivity (at the carbinolic center) determined by chromatographic separation. [c] With protocol A. [d] Using protocol B.

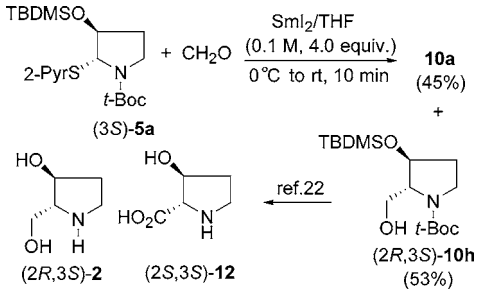
by HPLC analysis; however, its ^1H (500 MHz) and ^{13}C NMR (125 MHz) spectra (measured at room temperature in $CDCl_3$) clearly indicated the presence of two isomers. Keeping in mind that extensive rotamerism exists in carbamates such as **10a**, and to verify the nature of the isomerism of **10b**, ^1H and ^{13}C NMR experiments of **10b** were performed in deuterated DMSO at a range of temperatures. Indeed, at 25

°C, the ^{1}H and ^{13}C spectra of **10b** showed the presence of two isomers as did those recorded in $CDCl_3$ at 25 °C. At 80 °C, however, the ^{1}H and ^{13}C NMR spectra of **10b** in deuterated DMSO simplified and only one isomer was observed. When the temperature was lowered to 25 °C, the ^{1}H and ^{13}C NMR spectra of **10b** were identical with those taken prior to heating. These experiments clearly demonstrated that the isomers observed at room temperature were rotamers instead of diastereomers. Thus, the reductive *N*-α-hydroxyalkylation of **5a** led to only one isolable diastereomer. Consequently, the reductive hydroxyalkylation of **5a** was highly diastereoselective in establishing the C-2 chiral center of the pyrrolidine ring. As regarding the stereochemistry of the product, although the flexibility of the pyrrolidine ring does not allow the determination of stereochemistry, we have assumed that the *trans*-isomer is formed for steric reasons. This assumption was confirmed by the α-hydroxymethylation of **5a** with formaldehyde, which led to a known compound (*vide infra*).

We next examined the reductive couplings of **5a** with other ketones and aldehydes, and the results are summarized in Table 1. As can be seen, the reductive hydroxyalkylation of **5a** with symmetric ketones gave, in each case, only one diastereomer (Table 1, entries 2−5), and when unsymmetrical ketones or aldehydes were used, a diastereomeric mixture (originating from the newly formed exocyclic chiral carbinol center) was obtained (Table 1, entries 6−9). For ketones, protocol B generally gave better results than protocol A (Table 1, entries 2/3 and 6/7). The silyl group (TBDMS) protected pyridyl sulfide **5a** generally gave better results than the benzyl protected sulfide **5b**[21] did (Table 1, entries 3/10 and 8/11).

To illustrate the utility of the method, and to confirm the relative stereochemistry of the reaction, the reductive coupling of **5a** with formaldehyde was investigated (Scheme 4). The samarium(III) intermediate **D** reacted with formaldehyde to give **10h** as the only isolable diastereomer, which was identical in all respects with the known compound (2*R*,3*S*)-**10h**.[22] Since **10h** has been converted to (2*R*,3*S*)-2-hydroxymethyl-3-pyrrolidinol[23] (**2**) and its oxidized form, (2*S*,3*S*)-3-hydroxyproline[22] (**12**), a nonproteinogenic amino acid isolated from the hydrolysates of Mediterranean sponge,[24] and the seeds of *Delonix regia*,[25] our synthesis of **10h** thus constitutes formal asymmetric syntheses of **2** and **12**.[26] It is noteworthy that the present synthesis of **10h** also confirmed the 2,3-*trans*-diastereoselectivity of the reductive hydroxyalkylation of **5a** in the pyrrolidine ring.

Scheme 4. Formal Asymmetric Synthesis of Natural Products **2** and **12**



To summarize, through the present work, we were able to demonstrate that sulfide **5a** is a valuable synthetic equivalent of the samarium intermediate **D**, which can function synthetically as the hitherto unknown chiral nonracemic synthon **A**. Application of the present reductive coupling reaction to the asymmetric synthesis of more complex natural products is in progress.

Acknowledgment. The authors are grateful to the National Science Fund for Distinguished Young Investigators, the NSF of China (20272048; 203900505; 20402012), and the Ministry of Education (Key Project 104201) for financial support. We thank Professor J. M. Beau and Professor Y. M. Zhang for valuable discussions.

Supporting Information Available: Experimental procedures and spectral data for all new compounds and ^{1}H NMR and ^{13}C NMR spectra of compounds **5a**, **5b**, **7**, **8**, **10a**−**h**, **11a**, **11b**, and **11c**. This material is available free of charge via the Internet at http://pubs.acs.org.

OL047733Z

(20) All new compounds gave satisfactory analytical and spectral data.
(21) Sulfide **5b** was prepared in a similar way as that described for **5a**.
(22) Herdeis, C.; Hubmann, H. P.; Lotter, H. *Tetrahedron*: *Asymmetry* **1994**, *5*, 119.
(23) For other syntheses of (2*R*,3*S*)-2-hydroxymethyl-3-pyrrolidinol (**2**), see: (a) Hirai, Y.; Chintani, M.; Yamazaki, T.; Momose, T. *Chem. Lett.* **1989**, 1149. (b) Griffart-Brunet, D.; Langlois, N. *Tetrahedron Lett*. **1994**, *35*, 119. (c) Dell'Uomo, N.; Di Giovanni, M. C.; Misiti, D.; Zappia, G.; Delle Monache, G. *Tetrahedron: Asymmetry* **1996**, *7*, 181. (d) Barco, A.; Benetti, S.; De Risi, C.; Pollini, G. P.; Romagnoli, R.; Zanirato, V. *Tetrahedron Lett.* **1996**, *37*, 7599. (e) Aoyagi, Y.; Inaba, H.; Hiraiwa, Y.; Kuroda, A.; Ohta, A. *J. Chem. Soc., Perkin Trans. 1* **1998**, 3975. (f) Wee, A. G. H.; McLeod, D. D.; Rankin, T. J. *Heterocycles* **1998**, *48*, 2263. (g) Mascavage, L. M.; Lu, Q.; Vey, J.; Dalton, D. R.; Carroll, P. J. *J. Org. Chem.* **2001**, *66*, 3621. (h) Hulme, A. N.; Curley, K. S. *J. Chem. Soc., Perkin, Trans. 1* **2002**, 1083.
(24) (a) Irreverre, F.; Morita, K.; Robertson, A. V.; Witkop, B. *Biochem. Biophys. Res. Commun.* **1962**, *8*, 453. (b) Irreverre, F.; Morita, K.; Robertson, A. V.; Witkop, B. *J. Am. Chem. Soc.* **1963**, *85*, 2824. (c) Wolff, J. S.; Ogle, J. D.; Logan, M. A. *J. Biol. Chem*. **1966**, *241*, 1300.
(25) (a) Sung, M. L.; Fowden, L. *Phytochemistry* **1968**, *7*, 2061. (b) Szymanovicz, G.; Mercier, O.; Randoux, A.; Borel, J. P. *Biochimie* **1978**, *60*, 499.
(26) For other syntheses of (2*S*,3*S*)- or (2*R*,3*R*)-3-hydroxyproline (**12**) see: (a) Sheehan, J. C.; Whitney, J. G. *J. Am. Chem. Soc.* **1963**, *85*, 3863. (b) Thaning, M.; Wistrand, L. G. *Acta Chim. Scand.* **1989**, *43*, 290. (c) Hughes, P.; Clardy, J. *J. Org. Chem.* **1989**, *54*, 3260. (d) Mulzer, M. A.; Meier, A.; Bushmann, J.; Luger, P. *J. Org. Chem.* **1996**, *61*, 566. (e) Durand, J. O.; Larchevêque, M.; Petit, Y. *Tetrahedron Lett.* **1998**, *39*, 5743. (f) Poupardin, O.; Greck, C.; Genêt, P. *Synlett* **1998**, 1279. (g) Lee, J. H.; Kang, J. E.; Yang, M. S.; Kang, K. Y.; Park, K. H. *Tetrahedron* **2001**, *57*, 10071. (h) Huang, P.-Q.; Huang, H.-Y. *Synth. Commun.* **2004**, *34*, 1377.