Table II. Determination of ee's of Optically Active Amines

amine	% ee by weight	% ee by rotation	% ee by ^{31}P NMR
1. *l*-α-phenylethylamine	50	48[a]	50
2. *l*-α-phenylethylamine		95[a]	>98[b]
3. *l*-α-naphthylethylamine	33.3	32	34
4. *d*-α-naphthylethylamine		98	96
5. *d*-β-(*p*-methoxyphenyl)-α-phenylethylamine		*c*	94
6. *l*-valine methyl ester	82	*d*	84
7. *l*-valine methyl ester		100	>98[b]
8. *l*-phenylalanine methyl ester	80	*d*	77.5
9. *l*-homomethionine methyl ester	24	20	25
10. *l*-allylglycine methyl ester		96.6	97

[a] Commercially available, unpurified amine. [b] Other enantiomer could not be detected. [c] $[\alpha]_{578}$ + 64.3° (*c* 1.07, CH_3OH), ee = 95%.[9] [d] Not measured.

observed. Alkylphosphonothioic dichlorides have so far not been satisfactory for the ee determination of chiral secondary amines.

Several methods exist for the ee determination of amino acid derivatives and amines, e.g., chromatographic techniques using chiral phases[12] and NMR methods.[13] The new method presented here compares favorably with the other currently available techniques in view of the large shift differences obtained for the diastereoisomers, the simple experimental procedure (no workup), and the fact that in contrast to all other methods *no chiral auxiliary compound* is necessary.

A typical experimental procedure follows: To a stirred solution of primary amine or amino acid methyl ester (1.0 mmol) and triethylamine (1.0 mmol) and 1 mL of $CDCl_3$ was added at −20 °C 0.5 mmol of $MePSCl_2$ dissolved in 1 mL of $CDCl_3$. After being stirred for 10 min, the solution was transferred into a 10-mm NMR tube and the ^{1}H decoupled ^{31}P NMR spectrum recorded.

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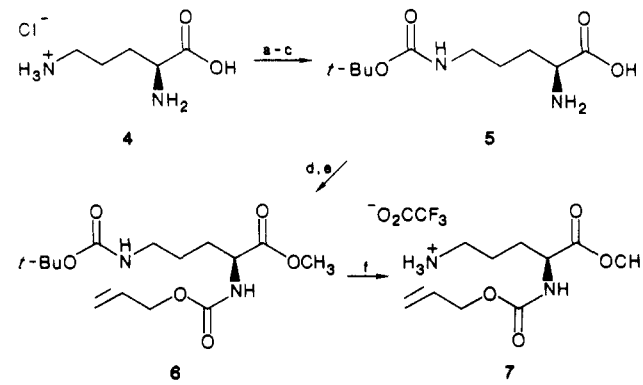
Studies Directed toward the Synthesis of Naturally Occurring Acyltetramic Acids. 2. Preparation of the Macrocyclic Subunit of Ikarugamycin

Summary: The synthesis of a model for the macrocyclic lactam system present in ikarugamycin (1) is described herein. The convergent route is culminated by a novel thermal intramolecular ketene trapping reaction with an amine to afford a 16-membered ring lactam. Treatment of lactams **21** and **22** with potassium *tert*-butoxide gave the model tetramic acids **3** and **23** via a transannular Dieckmann condensation.

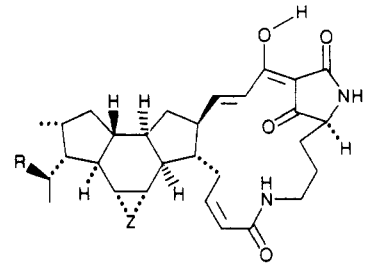
Sir: The antiprotozoal antibiotic ikarugamycin (1), a white crystalline substance isolated from the culture media of *Streptomyces phaeochromogenes*, and the structurally related compound capsimycin (2) are examples of a relatively rare macrocyclic lactam sytem containing a tetramic acid unit as well as a nonterpenoid tricarbocycle.[1]

Scheme I[a]

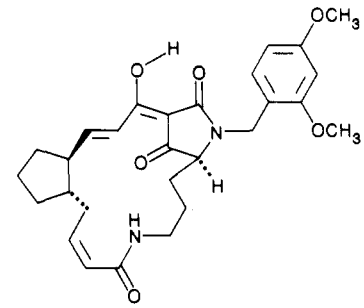


[a] Reagents: (a) $CuCO_3$ (1 equiv), H_2O, Δ, 0.75 h; (b) $((CH_3)_3C-O_2C)_2O$ (excess), $NaHCO_3$, H_2O–dioxane (3:1), 8 °C, 48 h; (c) H_2S; (d) $CH_2{=}CHCH_2OCOCl$ (excess), $NaHCO_3$, H_2O–dioxane (4:1), 0 °C, 16 h; (e) DCC (1.2 equiv), CH_3OH (excess), DMAP (catalytic), CH_2Cl_2, 0 °C, 1.5 h; (f) CF_3CO_2H–CH_2Cl_2 (1:3), 0 °C, 0.5 h.

We have previously reported the synthesis of the carbocyclic portion of 1 via an intramolecular [4 + 2] cycloaddition strategy.[2] The focus of our work has most recently been on the synthetically challenging macrocyclic lactam substructure.



1, Z = —CH=CH—, R = H
2, Z = O, R = OCH_3



3

Retrosynthetically, the macrocyclic unit may be viewed as comprising an L-ornithine subunit and a β-keto ester group bridged by a suitably substituted cyclopentane ring. Our convergent strategy called for the three units to be assembled followed by a macrocyclization (vide infra). The model study described below details the preparation of macrocycles **3** and **23** where the tricyclic system of 1 has been replaced by a single five-membered ring.

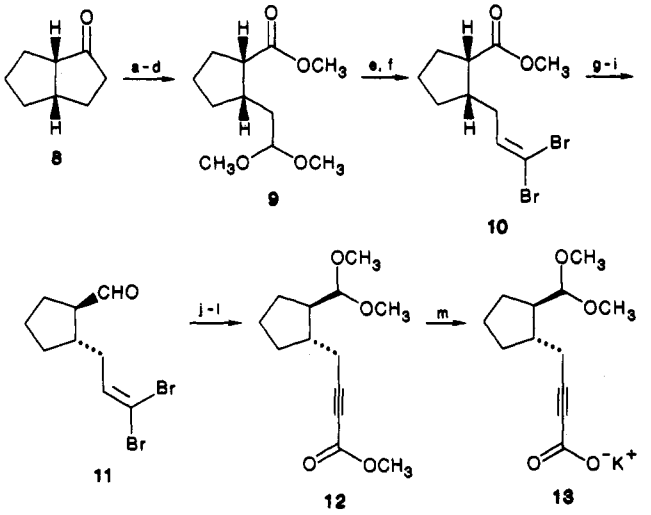
Construction of the requisite ornithine derivative **7** and cyclopentane unit **13** is described in Schemes I and II,

(1) (a) Jomon, K.; Kuroda, Y.; Ajisaika, M.; Saki, H. *J. Antibiot.* **1972**, *25*, 271. (b) Ito, S.; Hirata, Y. *Bull. Chem. Soc. Jpn.* **1977**, *50*, 1813. (c) Ito, S.; Hirata, Y. *Ibid.* **1977**, *50*, 227. (d) Ito, S.; Hirata, Y. *Tetrahedron Lett.* **1972**, 1181. (e) Ito, S.; Hirata, Y. *Ibid.* **1972**, 1185. (f) Ito, S.; Hirata, Y. *Ibid.* **1972**, 2557.

(2) Boeckman, R. K., Jr.; Napier, J. J.; Thomas, E. W.; Sato, R. I. *J. Org. Chem.* **1983**, *48*, 4152.

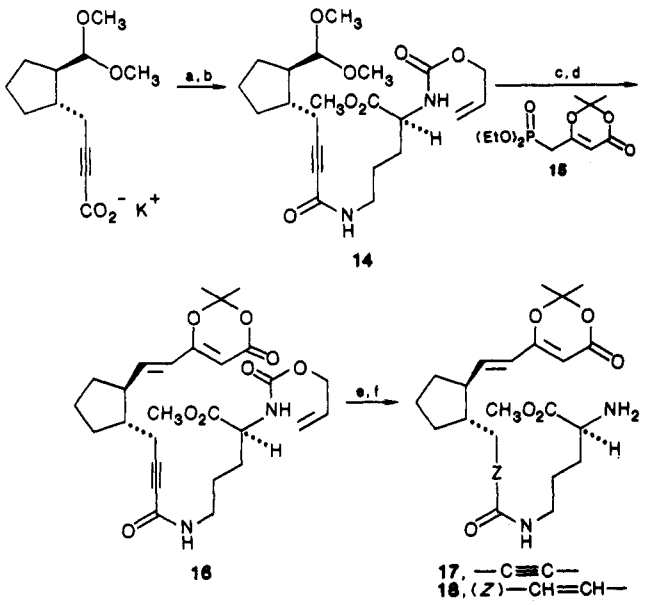
0022-3263/86/1951-5486$01.50/0

Scheme II[a]



[a] Reagents: (a) LDA (1.1 equiv), Me_3SiCl (1.1 equiv), THF, −78 °C, 1.33 h; (b) O_3, CH_3OH, CH_2Cl_2, −78 °C, 1.5 h; (c) $(CH_3)_2S$ (1 equiv), −78 → 25 °C, 12 h; (d) Amberlyst-15, CH_3OH (excess), Δ, 2 h; (e) Amberlyst-15, acetone–H_2O (95:5), 25 °C, 24 h; (f) CBr_4 (2 equiv), Ph_3P (4 equiv), CH_2Cl_2, 0 °C, 10 min; (g) *i*-Bu_2AlH (excess), hexane–Et_2O, 0 °C, 3 h; (h) PDC (excess), 3 Å sieves, CH_3CO_2H (catalytic), CH_2Cl_2, 25 °C, 0.75 h; (i) DBU (catalytic), CH_2Cl_2, 0 °C, 20 h; (j) CH_3OH (excess), Amberlyst-15, 25 °C, 2 h; (k) *n*-BuLi (2 equiv), THF, −78 (1 h) → 25 °C (1 h); (l) $ClCO_2CH_3$ (excess), THF, −78 → 25 °C, 16 h; (m) K_2CO_3 (1 equiv), CH_3OH–H_2O (5:3), 50 °C, 3 h.

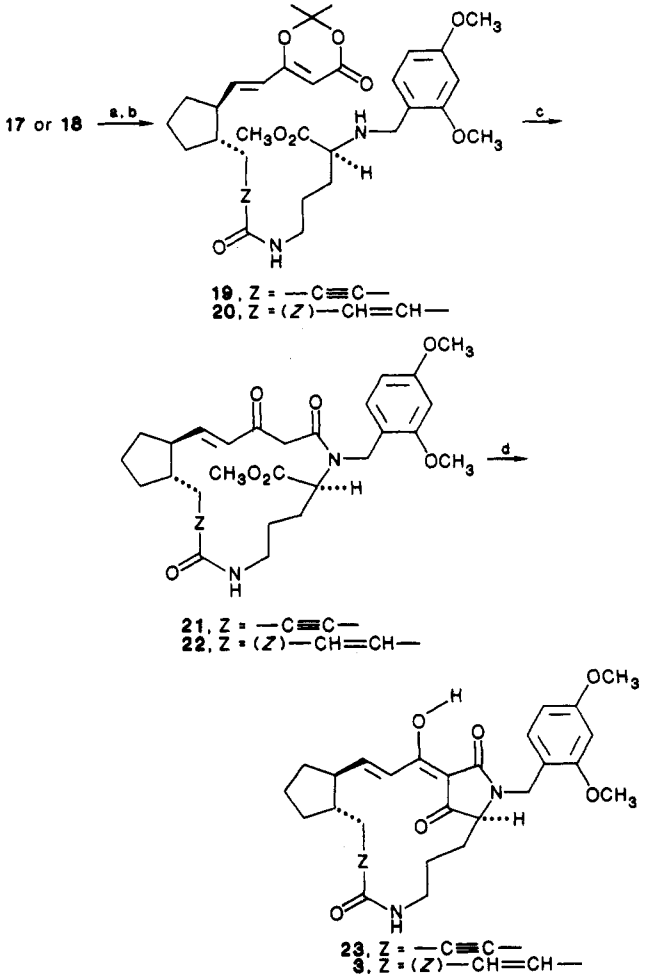
Scheme III[a]



[a] Reagents: (a) 2,4,6-$(CH_3)_3PhSO_2Cl$ (1.05 equiv), THF, 25 °C, 5 min; (b) **7** (1.2 equiv), DMAP (2.4 equiv), THF, 25 °C, 12 h; (c) Amberlyst-15, acetone–H_2O (95:5), 25 °C, 20 h; (d) **15** (1.1 equiv), *t*-BuOK (1.1), THF, 0 °C → 25 °C, 12 h; (e) $(PPh_3)_4Pd$ (catalytic), Ph_3P (catalytic), CH_3CO_2H (excess), CH_2Cl_2, 25 °C, 2 h; (f) H_2 (1 atm), 5% Pd–$BaSO_4$, quinoline (catalytic), CH_3CO_2H (catalytic), EtOAc, 25 °C, 1.5 h.

respectively.[3] The fully protected ornithine derivative **6** was easily assembled in three steps. Ornithine hydrochloride (**4**)[4] was converted to δ N-*t*-Boc protected amino acid **5** (mp 100–101 °C) via the copper complex.[5] Reaction of **5** with excess allyl chloroformate followed by esterification under standard conditions afforded **6** ($[\alpha]^{24}_D$ +1.40° (*c* 0.11, $CHCl_3$)) in 46% yield from **4**. The choice of the allyl carbamate for protection of the α-amino group was based on constraints imposed by the latter stages of the sequence which required a protecting group removable under essentially neutral conditions. Deprotection of the γ-amino group of **6** with trifluoroacetic acid (TFA) gave the amine salt **7** which was used without purification.

Scheme IV[a]



[a] Reagents: (a) 2,4-$(CH_3O)_2PhCHO$ (1 equiv), water removal (in vacuo), 25 °C, 16 h; (b) $NaCNBH_3$ (excess), CH_3OH, 25 °C, 2 h; (c) $PhCH_3$, Δ, 5 h; (d) *t*-BuOK (1 equiv), THF, 25 °C, 16 h.

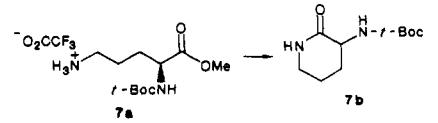
The sequence to the disubstituted cyclopentane **13** was somewhat more lengthy but nevertheless straightforward. Bicyclic ketone **8**[7] was converted to the dimethyl acetal **9** in good yield via ozonolysis of the related kinetic enol silyl ether and protection with methanol/acid. Deprotection

(3) The substances depicted in Scheme II are racemic materials. The substances depicted in Schemes III and IV are mixtures (~1:1) of optically active diastereomers. Only one diastereomer (that corresponding to **1**) is rendered in the interest of clarity and for convenience.

(4) L-(+)-Ornithine hydrochloride was obtained from the Aldrich Chemical Co.

(5) (a) Tesser, G. I.; Schwyzer, R. *Helv. Chim. Acta* **1966**, *49*, 1013. (b) Neuberger, A.; Sanger, F. *J. Biochem.* **1943**, *37*, 515. (c) Yajima, H.; Watanabe, H.; Okamoto, M. *Chem. Pharm. Bull.* **1971**, *19*, 2185.

(6) Neutralization of **7a** gave lactam **7b** in quantitative yield. Lactam formation is rapid with the conversion being complete in <5 min.



(7) Boeckman, R. K., Jr. *Tetrahedron Lett.* **1977**, 4281.

of acetal ester **9** with Amberlyst-15 followed by Wittig olefination afforded the cis dibromo olefin **10**.[8] Reduction of **10** with i-Bu_2AlH, reoxidation with PDC, and equilibration of the resulting cis aldehyde with DBU (catalytic) produced *trans*-**11** (19:1 t:c).[9] Aldehyde **11** was then reprotected as the dimethyl acetal and converted to acetylenic ester **12** (n-BuLi (2 equiv), THF, −78 °C, $ClCO_2Me$).[10] Hydrolysis of **12** smoothly afforded the required potassium salt **13** as a hygroscopic solid.

The coupling of the three segments and subsequent cyclization to the macrocycles are summarized in Schemes III and IV.[3] The assembly of three units could be initiated in a variety of ways. The sequence chosen appeared to be the most expedient since the formation of the sensitive and highly polar acyl tetramic acid unit was delayed until the final step.[11]

Treatment of **13** with 2,4,6-$(CH_3)_3PhSO_2Cl$ followed by in situ reaction of the resulting mixed anhydride with **7** in the presence of 4-(dimethylamino)pyridine (DMAP) (2 equiv) provided amide **14** (89%) as a mixture of diastereomers.[12-14] Acetal hydrolysis[8] followed by condensation with phosphonate **15**[15] afforded exclusively *trans*-**16** (62%).[12,16]

Treatment of **16** with $(Ph_3P)_4Pd$ (catalytic) cleaved the allyl carbamate under mild neutral conditions as required to furnish amino ester **17** (70% yield).[17] Reduction of the acetylenic linkage to the cis olefin **18** (~100% yield) was effected by a controlled hydrogenation (1 atm) over 5% Pd–$BaSO_4$ poisoned by quinoline.[12,18]

It proved necessary at this point to convert amines **17** and **18** to the corresponding N-2,4-dimethoxybenzyl derivatives (Scheme IV).[19,20] Condensation of **17** or **18** with 2,4-dimethoxybenzaldehyde in benzene followed by concentration in vacuo and reduction with $NaCNBH_3$ produced the secondary amines **19** and **20** (35 and 40% respectively, unoptimized).[12]

Successive ring closures were now required to complete construction of the required subunits **23** and **3**. Due to difficulties associated with manipulation of the tetramic acids, we elected to effect macrocyclization prior to the final Dieckmann condensation.[21] Cyclization of **19** and **20** occurs smoothly upon heating a dilute toluene solution (<0.01 M) at 110 °C for ~5 h, affording macrolactams **21** and **22** (57 and 80% respectively).[12] This new method of macrolactam formation proceeds with remarkable efficiency, presumably via the intermediacy of the acyl ketene.[22,23]

Facile Dieckmann cyclization of **21** and **22** ensued upon treatment with t-BuOK (1 equiv) in THF providing the target tetramic acids **23** (70%, λ_{max} 324 nm) and **3** (88%, λ_{max} 320 nm).[12,24,25]

The application of this methodology to the preparation of ikarugamycin (**1**) is currently under study.

Acknowledgment. This investigation was supported by grants from the National Science Foundation (CHE-81-19823) and the National Cancer Institute of the Na-

(8) Coppola, G. M. *Synthesis* **1984**, *12*, 1021.

(9) Czernecki, S.; Georgoulis, C.; Stevens, C. L.; Vijayakumaran, K. *Tetrahedron Lett.* **1985**, *26*, 1699. Use of this modification of the PDC oxidation resulted in greatly increased yields and reduced reaction times.

(10) Corey, E. J.; Fuchs, P. L. *Tetrahedron Lett.* **1972**, 3769.

(11) Two of the most obvious strategies employing macrolactam formation and macrocyclization by a Wadsworth–Emmons reaction were examined in preliminary studies. However, both were rendered unworkable by difficulties involved in manipulation of the highly functionalized acyl tetramic acid intermediates.

(12) All new substances exhibited IR, ^1H NMR (300 MHz), and mass spectral data consistent with the assigned structure and possessed satisfactory combustion or high resolution mass spectral analytical data. ^1H NMR data (δ at 300 MHz, $CDCl_3$). **3** (diastereomer 1): 7.19 (d, J = 8 Hz, 1 H), 7.12 (d, J = 16 Hz, 1 H), 6.76 (dd, J_1 = 16 Hz, J_2 = 10 Hz, 1 H), 6.43 (d, J = 16 Hz, 1 H), 6.42 (s, 1 H), 6.10 – 5.85 (m, 1 H), 5.83–5.65 (m, 2 H), 4.87 (dd, J_1 = 16 Hz, J_2 = 12 Hz, 2 H), 3.78 (s, 6 H), 3.70–3.37 (m, 3 H), 2.40–1.20 (m, 14 H). **3** (diastereomer 2): 7.25 (m, 1 H), 7.19 (d, J = 8 Hz, 1 H), 6.98 (d, J = 16 Hz, 1 H), 6.43 (d, J = 16 Hz, 1 H), 6.42 (s, 1 H), 6.10–5.85 (m, 1 H), 5.83–5.65 (m, 2 H), 4.16 (d, J = 15 Hz, 2 H), 3.78 (s, 6 H), 3.70–3.37 (m, 3 H), 2.40–1.20 (m, 14 H). **14**: 5.95–5.80 (m, 1 H), 5.28 (d, J = 17 Hz, 1 H), 5.19 (d, J = 10 Hz, 1 H), 4.55 (d, J = 5 Hz, 2 H), 4.40–4.26 (m, 1 H), 4.12 (d, J = 6 Hz, 1 H), 3.71 (s, 3 H), 3.33 (s, 3 H), 3.28 (s, 3 H), 2.60–2.16 (m, 2 H), 1.98–1.30 (m, 12 H). **16**: 6.37 (dd, 1 H), 5.95–5.76 (m, 2 H), 5.33 (d, J = 19 Hz, 1 H), 5.30 (s, 1 H), 5.14 (d, J = 10 Hz, 1 H), 4.59 (d, J v 5 Hz, 2 H), 4.44–4.30 (m, 1 H), 3.77 (s, 3 H), 3.36–3.26 (m, 2 H), 2.52–2.16 (m, 3 H), 2.09–1.40 (m, 11 H), 1.75 (s, 6 H). **17**: 6.45 (dd, 1 H), 5.96 (d, J = 15 Hz, 1 H), 5.30 (s, 1 H), 3.76 (s, 3 H), 3.54–3.44 (m, 1 H), 3.38–3.25 (m, 2 H), 2.52–2.10 (m, 3 H), 2.10–1.40 (m, 11 H), 1.74 (s, 6 H). **18**: 6.46–6.36 (dd, 1 H), 5.97–5.75 (m, 1 H), 5.86 (d, J = 16 Hz, 1 H), 5.63 (d, J = 12 Hz, 1 H), 5.20 (s, 1 H), 3.75 (s, 3 H), 3.48–3.40 (m, 1 H), 3.38–3.20 (m, 2 H), 2.77–2.67 (m, 1 H), 2.30–2.15 (m, 2 H), 1.98–1.20 (m, 11 H), 1.68 (s, 6 H). **19**: 7.10 (d, J = 10 Hz, 1 H), 6.48–6.38 (m, 3 H), 5.93 (d, J = 20 Hz, 1 H), 5.27 (m, 1 H), 3.83 (s, 3 H), 3.80 (s, 3 H), 3.70 (d, 2 H), 3.64 (s, 3 H), 3.39–3.20 (m, 3 H), 2.48–2.17 (m, 3 H), 2.00–1.40 (m, 11 H), 1.78 (s, 6 H). **20**: 7.12 (d, J = 10 Hz, 1 H), 6.55–6.41 (m, 3 H), 6.00–5.83 (m, 2 H), 5.58 (d, J = 12 Hz, 1 H), 5.17 (s, 1 H), 3.83 (s, 3 H), 3.81 (s, 3 H), 3.77 (m, 2 H), 3.75 (s, 3 H), 3.33–3.20 (m, 3 H), 2.83–2.76 (m, 2 H), 2.28–2.10 (m, 1 H), 2.00–1.23 (m, 11 H), 1.78 (s, 6 H). **21**: 7.19–7.00 (m, 2 H), 6.58–6.30 (m, 3 H), 4.80–4.40 (m, 2 H), 4.35–4.17 (m, 1 H), 3.83–3.75 (m, 9 H), 3.73–3.27 (m, 2 H), 2.70–1.20 (m, 16 H). **22**: 7.15 (d, J = 10 Hz, 1 H), 7.08 (d, J = 8 Hz, 1 H), 6.98–6.82 (m, 2 H), 6.50–6.20 (m, 3 H), 5.95–5.75 (m, 3 H), 5.65–5.46 (m, 1 H), 4.67–4.33 (m, 3 H), 3.82–3.75 (m, 9 H), 3.68–3.40 (m, 4 H), 2.40–2.00 (m, 2 H), 1.95–1.20 (m, 12 H). **23**: 7.19–7.10 (m, 1 H), 7.05–6.80 (m(br), 1 H), 6.45–6.30 (m, 2 H), 4.84 (d, J = 15 Hz, 2 H (1 diastereomer)), 4.18–4.05 (m, 2 H (1 diastereomer)), 3.78 (s, 6 H), 3.70–3.50 (m, 3 H), 2.75–1.20 (m, 14 H).

(13) Kitamura, M.; Isobe, M.; Ichikawa, Y.; Goto, T. *J. Am. Chem. Soc.* **1984**, *106*, 3252.

(14) The production of diastereomers in this case was inconsequential and no effort was made to effect a separation, since in the system leading to **1** both components in this coupling will be of the correct absolute configuration; see ref 2.

(15) (a) Boeckman, R. K., Jr.; Thomas, A. J. *J. Org. Chem.* **1982**, *47*, 2823. (b) Boeckman, R. K., Jr.; Perni, R. B.; McDonald, J. E.; Thomas, A. J. *Org. Synth.*, in press.

(16) In order to obtain reproducibly good yields (60–68%), this condensation had to be effected by slow addition of a THF solution of t-BuOK (1.1 equiv) to a THF solution of the aldehyde derived from **14** (1.0 equiv) and **15** (1.1 equiv) at 25 °C. Alternate methods for combining the reagents afforded variable yields which were distinctly inferior to those obtained by the above procedure.

(17) Jeffrey, P. D.; McCombie, S. W. *J. Org. Chem.* **1982**, *47*, 587.

(18) A catalytic amount of acetic acid was required to initiate the reaction. Omission of the quinoline resulted in mixtures of **17** and **18** for short reaction times or **18** and the saturated amide at longer reaction times. Reduction of the dienone was never observed.

(19) Repeated attempts to effect Dieckmann condensation on N-unsubstituted β-keto lactams related to **21** and **22** were unsuccessful. We, therefore, elected to employ a protecting group for nitrogen, see: (a) Schlessinger, R. H.; Bebernitz, G. R.; Lin, P.; Poss, A. J. *J. Am. Chem. Soc.* **1985**, *107*, 1777. (b) DeShong, P.; Ramesh, S.; Elango, V.; Perez, J. *J. Am. Chem. Soc.* **1985**, *107*, 5219. (c) Boeckman, R. K., Jr.; Starrett, J. E., Jr.; Nickell, D. G.; Sum, P.-E. *J. Am. Chem. Soc.* **1986**, *108*, 5549. Furthermore, we have demonstrated that **1** is stable to the conditions required for removal of this group: anhydrous CF_3CO_2H (in excess as solvent), 1 h, 25 °C.

(20) The assembly of a fully protected N-substituted ornithine derivative appears quite feasible and is currently under investigation. Thus, the required N-protection operation can be incorporated at an early stage without markedly affecting the overall efficiency of the sequence.

(21) Our experience during our studies of tetramic acids has repeatedly been that complex acyl tetramic acids are chemically sensitive, often not readily purified by conventional methods and difficult to characterize definitively by spectroscopic (NMR) techniques (due to spectral broadening possibly due to ion exchange phenomena). These problems are particularly prevalent in N-unsubstituted derivatives.

(22) Hyatt, J. A.; Feldman, P. L.; Clemens, R. J. *J. Org. Chem.* **1984**, *49*, 5105.

(23) This methodology is somewhat restricted in scope since β-keto lactams are produced, however, in many instances further transformation to α,β-unsaturated and saturated macrolactams will be readily accomplished. The method appears adaptable equally well to macrolide synthesis, and studies of this varient are in progress.

(24) (a) Lacy, R. N. *J. Chem. Soc.* **1954**, 580. (b) Lee, V. J.; Branfman, A. R.; Herrin, T. R.; Rinehart, K. L., Jr. *J. Am. Chem. Soc.* **1978**, *100*, 4225.

(25) Ikarugamycin (**1**) exhibits λ_{max} 327 nm, see ref 1b.

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Synthesis of Mugineic Acid through Direct C-Acylation Using Diphenyl Phosphorazidate[1,2]

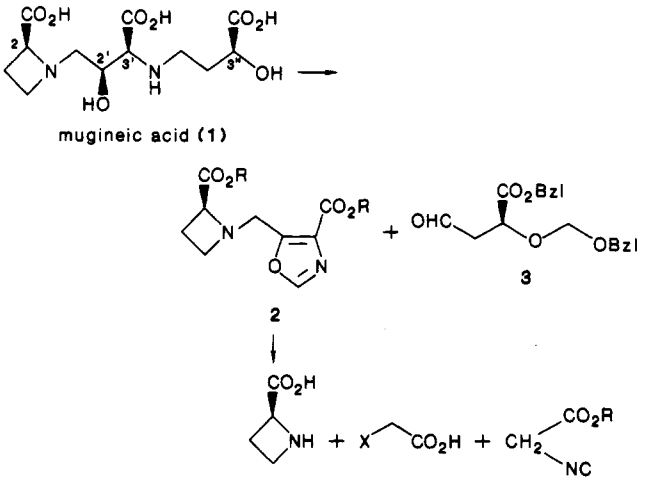
Summary: The first synthesis of mugineic acid, a typical phytosiderophore from roots of barley, has been achieved through direct C-acylation using diphenyl phosphorazidate (DPPA).

Sir: There has been considerable interest in new types of iron-chelating amino acids isolated from the root washings of gramineous plants.[3] They are called phytosiderophores, which promote uptake and transport of iron in higher plants. Mugineic acid[4] (1) is a typical phytosiderophore excreted from roots of barley. Its structure and iron transport mechanism have been well clarified by Nomoto and co-workers.[3a,c] Further investigations by Nomoto's group have recently revealed[5] that mugineic acid exerts an interesting inhibitory effect against angiotensin-converting enzyme. Although 2′-deoxymugineic acid, another phytosiderophore, was elegantly synthesized by Ohfune and co-workers,[6] there have been no reports to date on the synthesis of mugineic acid itself.

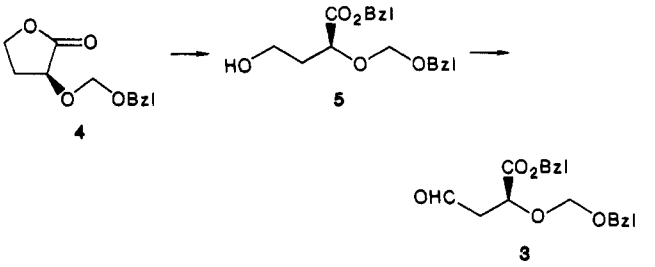
We now report the first synthesis of mugineic acid (1). Retrosynthetic analysis of 1, based on direct C-acylation using diphenyl phosphorazidate (DPPA, $(C_6H_5O)_2P(O)N_3$) recently developed by our group,[7] revealed two building blocks as shown in Scheme I. One is the 4-(alkoxycarbonyl)oxazole 2 which will serve as a latent erythro β-hydroxy-α-amino acid[8] and be synthesized from (*S*)-azetidine-2-carboxylic acid, haloacetic acid, and isocyanoacetate. The other is the (*S*)-malic acid half-aldehyde derivative 3 which will constitute the right-half fragment of 1.

The known γ-lactone[9] 4 was converted to 3 in three steps as shown in Scheme II.[10] Hydrolysis of 4 with aqueous potassium hydroxide (1 equiv) in dimethylformamide–water (4:1) (room temperature, 2 h), followed by esterification with a mixture of benzyl bromide (1.5 equiv), 18-crown-6 (0.1 equiv), and potassium bicarbonate (1 equiv) (room temperature, 18 h), afforded the benzyl ester 5 as a colorless oil, $[\alpha]^{21}_D$ –61.8° (*c* 1, CH_2Cl_2). Treatment of 5 with sulfur trioxide–pyridine complex (3 equiv) and triethylamine (6 equiv) in dimethyl sulfoxide–methylene chloride (1:1) (10 °C, 30 min) gave the aldehyde 3 as a colorless oil, $[\alpha]^{23}_D$ –45.3° (*c* 1, CH_2Cl_2), in 70% yield. The

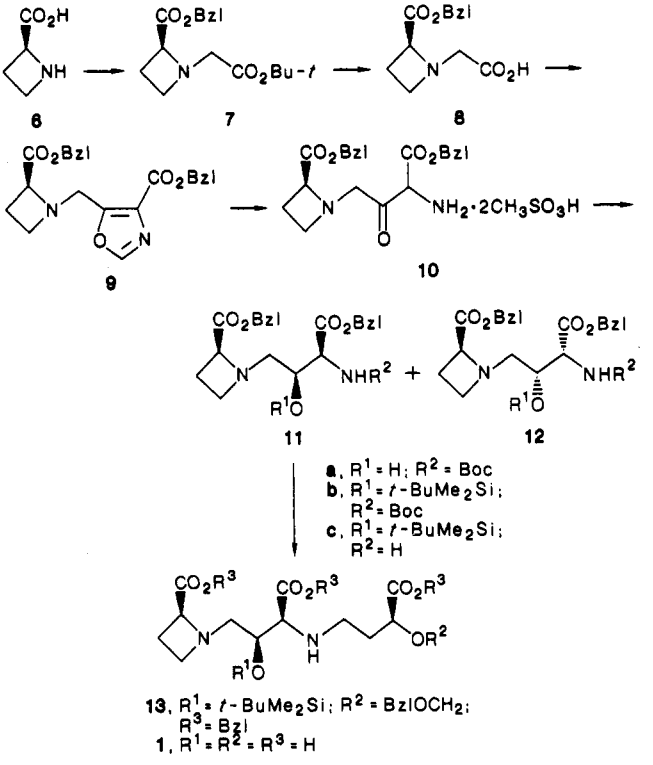
Scheme I



Scheme II



Scheme III



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(10) All of the products gave satisfactory IR, NMR, and high resolution mass spectra.

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