7.3–7.8 (m, 13, Ar Hs), 8.1–8.3 (m, 4, Ar Hs); exact mass, *m/e* 307.1361 (calcd for $C_{23}H_{17}N$, *m/e* 307.1361).

2,6-Diphenyl-4-*n*-butylpyridine (2c) was prepared from a 2.00 M ethereal solution of *n*-butylmagnesium bromide, **1a**, and 27.1 mg (0.050 mmol) of dpppNiCl$_2$: liquid; UV λ_{max} 246 nm (log ϵ 4.43); IR 1595 (m, C=C), 1580 (m), 1540 (m) cm^{-1}; ^{1}H NMR δ (CCl_4) 0.95 (t, 3, J = 7 Hz, Me), 1.2–1.9 (m, 4, methylenes), 2.68 (t, 2, J = 7 Hz, benzyl Hs), 7.3–7.5 (m, 8, Ar Hs), 8.0–8.2 (m, 4, Ar Hs); exact mass, *m/e* 287.1674 (calcd for $C_{21}H_{21}N$, *m/e* 287.1674).

2,6-Diphenyl-4-cyclohexylpyridine (2d) was prepared from a 2.00 M ethereal solution of cyclohexylmagnesium bromide, **1a**, and 27.1 mg (0.050 mmol) of dpppNiCl$_2$: liquid; UV λ_{max} 246 nm (log ϵ 4.47); IR 1600 (m, C=C), 1580 (m), 1550 (m) cm^{-1}; ^{1}H NMR δ (CCl_4) 1.2–2.1 (m, 10, methylenes), 2.3–2.7 (m, 1, methine), 7.3–7.5 (m, 8, Ar Hs), 8.1–8.3 (m, 4, Ar Hs); exact mass, *m/e* 313.1830 (calcd for $C_{23}H_{23}N$, *m/e* 313.1830).

2,6-Diphenylpyridine (2e) was prepared from a 2.00 M ethereal solution of cyclohexylmagnesium bromide and **1a**: mp 80–81 °C (EtOH) (lit.[13] mp 81–82 °C); UV λ_{max} 246 nm (log ϵ 4.42); IR 1590 (m, C=C), 1560 (m), 1540 (m) cm^{-1}; ^{1}H NMR δ (CCl_4) 7.3–7.6 (m, 9, Ar Hs), 8.0–8.2 (m, 4, Ar Hs); exact mass, *m/e* 231.1048 (calcd for $C_{17}H_{13}N$, *m/e* 231.1048).

2,6-Bis(*p*-methoxyphenyl)-4-methylpyridine (2f) was prepared from a 2.85 M ethereal solution of methylmagnesium bromide and **1b**: mp 121 °C (hexane); ^{1}H NMR δ ($CDCl_3$) 2.43 (s, 3, Me), 3.89 (s, 6, 2 OMe), 7.04, 8.12 (d, 2 each, J = 9 Hz, Ar Hs), 7.41 (s, 2, pyridine β-Hs).

Anal. Calcd for $C_{20}H_{19}O_2N$: C, 78.65; H, 6.28; N, 4.59. Found: C, 78.73; H, 6.30; N, 4.56.

2,6-Di-2-furyl-4-methylpyridine (2g) was prepared from a 2.85 M ethereal solution of methylmagnesium bromide and **1c**: mp 64 °C (hexane); ^{1}H NMR δ ($CDCl_3$) 2.45 (s, 3, Me), 6.5–6.7 (m, 2, furan 2 H-4), 7.15 (d, 2, J = 5 Hz, 2 H-3), 7.44 (s, 2, pyridine β-Hs), 7.59 (d, 2, J = 1 Hz, 2 H-5).

Anal. Calcd for $C_{14}H_{11}O_2N$: C, 74.64; H, 4.93; N, 6.22. Found: C, 74.57; H, 4.97; N, 6.20.

2,6-Di-2-thienyl-4-methylpyridine (2h) was prepared from a 2.85 M ethereal solution of methylmagnesium bromide and **1d**: mp 81 °C (hexane); ^{1}H NMR δ ($CDCl_3$) 2.42 (s, 3, Me), 7.1–7.7 (m, 6, thiophene Hs), 7.36 (s, 2, pyridine β-Hs).

Anal. Calcd for $C_{14}H_{11}NS_2$: C, 65.32; H, 4.32; N, 5.44. Found: C, 65.21; H, 4.36; N, 5.40.

4-Methyl-2-phenyl-6-(2-thienyl)pyridine (2i) was prepared from a 2.85 M ethereal solution of methylmagnesium bromide and **1e**: mp 102–104 °C (Et_2O); ^{1}H NMR δ ($CDCl_3$) 2.43 (s, 3, Me), 7.1–7.7 (m, 3, thiophene Hs), 7.49 (s, 5, benzene Hs), 8.1–8.3 (m, 2, pyridine β-Hs).

Anal. Calcd for $C_{16}H_{13}NS$: C, 76.45; H, 5.22; N, 5.57. Found: C, 76.26; H, 5.29; N, 5.52.

α-(Methylthio)indole (4a) was prepared from thiooxindole (**3b**) by a published procedure.[8] The latter, in turn, was produced in the following manner.

A solution of 2.60 g (20.0 mmol) of oxindole (**3a**) in 20 mL of dry hexamethylphosphoramide was flushed with argon, and 4.00 g (10.0 mmol) of 2,4-bis(*p*-methoxyphenyl)-1,3,2,4-dithiadiphosphetane 2,4-disulfide[9] was added portionwise. The mixture was heated at 110 °C under argon for 1 h, and the orange solution was cooled and poured into 100 mL of water. It was extracted exhaustively with ether, and the extract was dried (Na_2SO_4) and evaporated. Chromatography of the residual oil on a short silica column and elution with 2:1 hexane–ethyl acetate gave 2.70 g (90%) of yellow, crystalline **3b**: mp 147–149 °C (lit.[8] mp 147–149 °C).

General Procedure for the Reactions of Grignard Reagents with α-(Methylthio)indole (4a). A 1.6 M hexane solution of *n*-butyllithium, 0.75 mL (1.20 mmol), was added to a solution of 200 mg (1.20 mmol) of α-(methylthio)indole (**4a**) in 10 mL of dry benzene, and the mixture was stirred under argon at room temperature for 15 min.

A 2.90 M ethereal solution of methylmagnesium bromide, 86 μL (0.25 mmol), was added dropwise to a stirring suspension of 70 mg (0.12 mmol) of dpppNiCl$_2$ in 5 mL of dry benzene under argon, and the mixture refluxed was for 15 min. It then was added to the above suspension of the lithio salt of **4a**. Immediately thereafter there was added 1.40 mmol of required Grignard reagent, and the mixture was heated at 80 °C for the time cited below. It then was cooled and poured into 50 mL of 1 N hydrochloride acid solution, and the water solution was extracted with ether. The combined organic solutions were washed with saturated brine solution, dried (K_2CO_3), and evaporated. The residue was crystallized or chromatographed on silica gel (elution with 20:1 hexane–ethyl acetate).

For the reactions utilizing the magnesio salt of **4a**, whose product yields are quoted in the Discussion section, the above *n*-butyllithium solution was replaced by 0.42 mL (1.20 mmol) of a 2.90 M ethereal solution of methylmagnesium bromide. The product yields of the reactions utilizing the lithio salt of **4a** were slightly lower from those of the reactions of the magnesio salt.

α-Phenylindole (4b) was prepared from a 2.80 M ethereal solution of phenylmagnesium bromide (3.5 h): mp 187–188 °C (lit.[14] mp 188–189 °C), spectrally identical with an authentic sample.

α-Methylindole (4c) was prepared from a 2.90 M ethereal solution of methylmagnesium bromide (6 h): mp 56–58 °C (lit.[15] mp 58–60 °C), spectrally the same as an authentic sample.

α-*n*-Butylindole (4d) and indole (4e) were prepared from a 2.80 M ethereal solution of *n*-butylmagnesium bromide (3.5 h). **4d**: IR 3400 (m, NH), 1620 (w), 1590 (w), 1550 (m, C=C); ^{1}H NMR δ (CCl_4) 0.86 (t, 3, J = 7 Hz, Me), 1.1–1.8 (m, 4, methylenes), 2.51 (t, 2, J = 7 Hz, benzyl CH_2), 6.09 (br s, 1, indole β-H), 6.6–7.6 (m, 5, Ar Hs, NH); spectrally identical with literature data.[16] **4e**: spectrally the same as an authetic specimen.

Indole (4e) was prepared from a 1.90 M ethereal solution of isopropylmagnesium bromide and 81 mg (0.12 mmol) of [(C_6H_5)$_3$P]$_2$NiCl$_2$ (replacing the above dpppNiCl$_2$) (45 h): **4e** spectrally identical with sample above.

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The Direct Cyclization of α-Acylamino-Substituted Hydroxamates to β-Lactams

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With the discovery of biologically active monocyclic β-lactam antibiotics such as the nocardicins **1**, monobactams **2**[1] (monosulfactams **3**), and, most recently, the oxamazins **4**,[2] considerable importance has been placed on the synthesis of such key intermediates as **5a** and **5b**. Prior routes to these molecules have utilized expensive reagents or resulted in competitive side-product formation, or both. Herein is provided a straightforward and efficient route to these versatile β-lactams (Scheme I).

By taking advantage of the low pK of the hydroxamate N–H bond (pK 6–10), we previously developed a synthetic route which relied on a Mitsunobu reaction[3] to promote the key cyclization step (eq 1, X = OH).[4] This proved effective when the nitrogen was protected as a carbamate (i.e., **6b**, **6c**). However, when R was a simple acyl derivative

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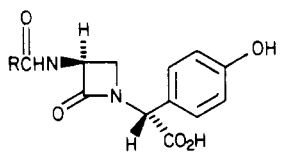
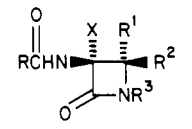
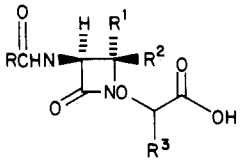


1



2, $R^3 = SO_3^-$; X = OCH_3
3, X = H; $R^3 = OSO_3^-$



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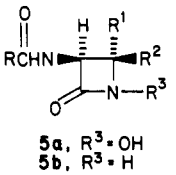
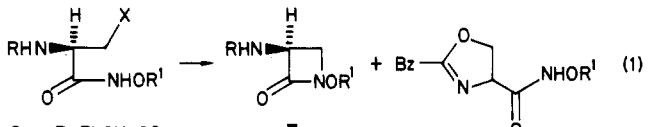


5a, R^3 = OH
5b, R^3 = H

(6a) direct cyclization to the β-lactam **7a** could not be effected without competitive oxazoline (**8a**) formation. Additionally, the difficulty of selective 3-amino deprotection of **7** led us to the development of alternate routes. A simplified approach was later developed (eq 1, X = OH,



(1)

6a, R = $PhCH_2CO$
6b, R = Cbz
6c, R = t-Boc

7

8a

R′ = acyl).[5] Although circumventing many of the other shortcomings of the previous route with normal acylamino substituents, oxazoline formation became even more predominant. We therefore turned to direct cyclization to β-lactams, without the requisite protection/deprotection steps.

In our very early work, we had demonstrated that β-chloro hydroxamates (eq 1, X = Cl) were readily converted to β-lactams by mild base treatment.[4] Subsequently, the Squibb group utilized β-mesylated amino acid derivatives (eq 1, **6c**, X = OMs) for cyclization directly to the monolactams.[6] In neither case had the cyclization reaction been reported for simple acylated amino acid derivatives.

Conceptually, base treatment of the mesylate **9** (Table I) could allow the formation of any one of four products: desired β-lactam **10**, oxazoline **11**, aziridine **12**, or the elimination product **13**. Indeed, initial attempts to cyclize the mesylate of (phenylacetyl)serine *O*-benzylhydroxamate **9a** were discouraging. As shown in Table I, under most conditions tried, the undesired products **11**, **12**, and **13** were formed in addition to the β-lactam **10**. However, we subsequently observed that careful treatment of the mesylate **9a** with 1 equiv of potassium *tert*-butoxide in DMF at −23 °C gave the desired β-lactam in 75% recrystallized yield, without the need for chromatography. TLC analysis of the reaction mixture showed none of the previously observed side products.

As a further demonstration of the utility of these cyclization conditions, the reaction was carried out on the mesylate of the biologically more interesting *syn*-2-(2-aminothiazol-4-yl)-2-(methoxyimino)acetyl (ATMO) side chain **9b**. Again, only desired β-lactam was detectable in the crude reaction mixture. Extractive workup followed by recrystallization gave **10b** in 40% isolated yield. ^1H NMR of **10b** indicated that the basic cyclization conditions did partially isomerize the oxime.

The mesylates **9a,b** were easily prepared from the corresponding alcohols in yields of 80–95%. The mesylates can be recrystallized but undero decomposition upon storage and therefore were carried directly on to the cyclization step without isolation. By employing an approximately 1 M solution of potassium *tert*-butoxide in *tert*-butyl alcohol, 1 equiv of base can be added without the introduction of moisture into the reaction. At temperatures lower than −23 °C, the potassium *tert*-butoxide begins to precipitate out of solution.

In summary, a straightforward and direct method of β-lactam formation has been developed, which allows for the cyclization of simple α-acylamino-substituted hydroxamates without competitive oxazoline formation. In addition to eliminating separate protection/deprotection steps, in the examples studied, no chromatographic purification of the products was necessary.

Experimental Section

General Comments. Melting points were taken on a Thomas-Hoover melting point apparatus and are uncorrected. IR spectra was recorded on a Perkin-Elmer 727b spectrometer. ^1H NMR spectra were obtained in $CDCl_3$ with Me_4Si as a reference, unless stated otherwise, on a Varian EM390 or Nicolet NB300 spectrometer. Mass spectra were recorded on an AEI Scientific Apparatus 902. Elemental analyses were performed by Midwest Microlabs, Indianapolis, IN, or M-H-W Laboratories, Phoenix, AZ. Thin-layer chromatography was run on Kieselgel 60 sheets using EtOAc/hexane solvent systems.

Compounds 14a,[4] 14c,[4] and 15a[7] were prepared according to previously published procedures.

***t*-BuO⁻K⁺/*t*-BuOH Solution.** An approximately 1 M solution was prepared by dissolving 2.4 g of potassium in 60 mL of *t*-BuOH and stirring for 48 h at room temperature under nitrogen. The solution was standardized by titration with 0.1043 M HCl solution using phenolphthalein as the indicator.

***N*-[*syn*-2-(2-Aminothiazol-4-yl)-2-(methoxyimino)acetyl]-L-serine *O*-Benzylhydroxamate (14b).** Compound **14d** (431 mg, 1.75 mmol) was dissolved in 10 mL of THF along with Et_3N (0.48 mL, 3.5 mmol) and cooled to −10 °C (ethanol/ice bath). While stirring, *N*-hydroxybenzotriazole *syn*-2-(2-aminothiazol-4-yl)-2-(methoxyimino)acetate[9] (837 mg, 2.63 mmol) was added portionwise. The reaction warmed to room temperature and stirred for 24 h. The solvent was evaporated and the remaining oil chromatographed (EtOAc/THF, 1:1). The desired product was recrystallized from ethanol/ether to give 333 mg (48%) of **14b**: mp 105 °C sinter and dec; NMR ($CDCl_3$ + MeOH-d_4) δ 8.1 (br s, 1 H), 7.38 (s, 5 H), 6.88 (s, 1 H), 6.45 (br s, 2 H), 4.9 (s, 2 H), 4.5 (m, 1 H), 4.0 (m, 1 H), 3.82 (s, 4 H); IR (KBr) 1650 cm^{-1}.

L-Serine *O*-Benzylhydroxamate *N*-Hydrochloride (14d). Compound **14c** (4.43 g, 14.3 mmol) was dissolved in 100 mL of EtOAc at room temperature. Dry HCl gas was bubbled through the stirred solution for a period of 1 h. After purging with nitrogen, the addition of ether further precipitated the product which was isolated by filtration in 93% yield: mp sinter 98–105 °C; NMR (D_2O) δ 7.33 (s, 5 H), 4.76 (s, 2 H), 3.5–3.8 (m, 3 H); IR (KBr) 1665, 1160 cm^{-1}. Anal. Calcd for $C_{10}H_{15}N_2O_3C$: C, 48.69; H, 6.13; N, 11.36. Found: C, 48.36; H, 6.27; N, 11.35.

***N*-(Phenylacetyl)-*O*-mesyl-L-serine *O*-Benzylhydroxamate (9a).** Alcohol **14a** (154 mg, 0.47 mmol) was dissolved in dry pyridine (5 mL) and cooled to 0 °C under nitrogen.

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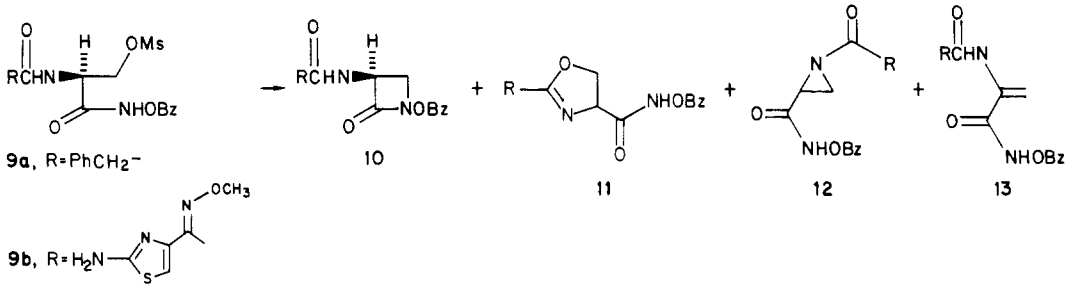
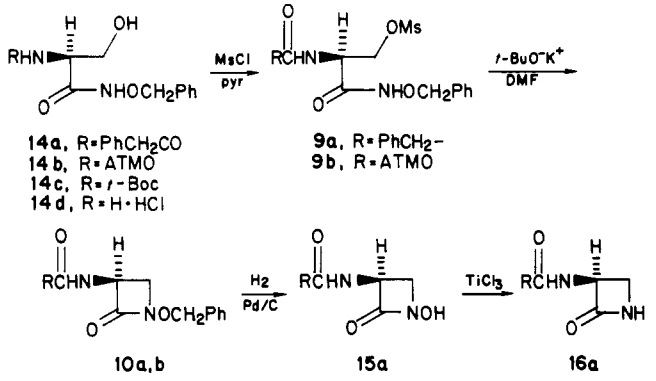
Table I



reactant	reaction conditions	product ratio, % 10	11	12	13
9a	$KHCO_3/CH_3CHCl_2$, Δ		94		
9a	*t*-BuO⁻K⁺/THF, Δ	26		47	9
9a	*t*-BuO⁻K⁺/THF, 0 °C	34		43	
9a	*t*-BuO⁻K⁺/DMF, 0 °C	55		21	
9a	*t*-BuO⁻K⁺/DMF, −23 °C	75			
9b	*t*-BuO⁻K⁺/DMF, −23 °C	40			

Scheme I



While stirring, methanesulfonyl chloride (0.044 mL, 0.56 mmol) was added, neat, by syringe. After 3 h the reaction mixture was poured into EtOAc (50 mL), washed with 1.2 N HCl until acidic and then with brine, and dried ($MgSO_4$), and the solvent was evaporated. The residue was redissolved in CH_2Cl_2 and eluted through a plug of silica gel. Solvent evaporation gave **9a** as a white solid in 95% yield (182 mg). Compound **9a** was once carefully recrystallized from EtOAc/hexanes in 82% yield, otherwise purification by recrystallization was not necessary: mp 94–96 °C; NMR ($CDCl_3$) δ 7.32 (s, 5 H), 7.22 (s, 5 H), 4.77 (s, 2 H superimposed on m, 1 H), 4.3 (m, 2 H), 3.38 (s, 2 H), 2.84 (s, 3 H); IR (KBr) 1620 br, 1160 cm^{-1}.

***N*-[*syn*-2-(2-Aminothiazol-4-yl)-2-(methoxyimino)acetyl]-*O*-mesyl-L-serine *O*-benzylhydroxamate (9b)** was prepared by the above mesylation procedure from **14b** in 62% yield (recrystallized from EtOAc/hexanes) except that the pyridine was removed by washing with a KH_2PO_4/H_3PO_4 buffer, pH 3.5: mp 96 °C sinter and dec; NMR ($CDCl_3$ + acetone-d_6) δ 8.5 (d, NH), 7.37 (s, 5 H), 6.90 (s, 1 H), 4.93 (s, 2 H), 4.3–5.0 (m, 3 H), 3.81 (s, 3 H), 3.06 (s, 3 H); IR (KBr) 1760, 1165, 1030 cm^{-1}.

1-(Benzyloxy)-3-(phenylacetamido)-2-azetidinone (10a). Mesylate **9a** (924 mg, 2.27 mmol) was dissolved in DMF (20 mL, shaken with KOH and freshly distilled over CaO) and cooled to −23 °C (CCl_4/CO_2) under nitrogen. While stirring, *t*-BuO⁻K⁺/*t*-BuOH solution (2.10 mL, 1.08 M, 2.27 mmol) was added, and the reaction was allowed to slowly warm to room temperature overnight. After 10 h the mixture was poured into EtOAc (100 mL) and washed twice with 5% $NaHCO_3$ (25 mL) and then several times with water to remove the DMF. The organic layer was dried ($MgSO_4$) and the residue was recrystallized from EtOAc/hexanes to give **10a** in 75% yield (529 mg): mp 124–126 °C (note: depending on the method of preparation, differing melting points have been reported for this compound, both in our laboratories (127.5–129 °C)[4] and those of Squibb (126–128 °C, 130–131 °C)[7]); NMR ($CDCl_3$) δ 7.41 (s, 5 H), 7.30 (s, 5 H), 6.5 (br s, 1 H), 4.94 (s, 2 H), 4.65 (m, 1 H), 3.54 (s, 2 H superimposed on t, 1 H), 3.15 (dd, 1 H); IR (KBr) 1760, 1650 cm^{-1}; IR ($CHCl_3$) 1780, 1680 cm^{-1}; this material was identical with previously reported material.[4]

1-(Benzyloxy)-3-[*syn*-2-(2-aminothiazol-4-yl)-2-(methoxyimino)acetamido]-2-azetidinone (10b) was prepared by the above cyclization procedure from **9b** and recrystallized from EtOAc/hexanes in 40% yield as a mixture of isomers: NMR (acetone-d_6) δ 8.63 (d, NH), 7.44 (s, 5 H), 6.88 and 6.73 (s, 1 H, isomeric), 5.9 (br s, 2 H), 5.00 (s, 2 H), 4.67 (m, 1 H), 4.02 and 3.90 (s, 3 H, isomeric), 3.70 (apparent t, 1 H), 4.46 (dd, 1 H); IR (KBr) 1760, 1650 cm^{-1}.

3-(Phenylacetamido)-2-azetidinone (16a) was prepared form **15a** according to previously published reduction procedures[8] with the following modification: Prior to basification and workup, tartaric acid (250 mol % relative to $TiCl_3$) was added to facilitate Ti^{IV} removal. The pH was then adjusted to 8 with 10% Na_2CO_3 and worked up as reported. Recrystallization from EtOAc/hexanes gave **16a** in 35% yield: mp 166–168 °C dec; NMR ($CDCl_3$ + Me_2SO-d_6) δ 8.25 (br s, 1 H), 7.26 (s, 5 H), 4.98 (m, 1 H), 3.52 (s, 2 H superimposed on t, 1 H), 3.2 (dd, 1 H); IR (KBr) 1770 cm^{-1}. Anal. Calcd for $C_{11}H_{12}N_2O_2$: C, 64.69; H, 5.92; N, 13.72. Found: C, 64.44; H, 5.77; N, 13.47.

Acknowledgment. We are grateful for the support of our research by the NIH, Eli Lilly and Company, and a Reilly Fellowship for M.A.K. The 300-MHz NMR system used was obtained by grants from the NIH and the University of Notre Dame. Ms. Therese Debiak and Mrs. Kathleen Peterson recorded the 300-MHz NMR spectra.

Registry No. 9a, 95070-21-6; **9b**, 95070-22-7; **10a**, 75624-37-2; **10b** (isomer 1), 95070-23-8; **10b** (isomer 2), 95070-24-9; **11a**, 95070-25-0; **12a**, 95070-26-1; **13a**, 95070-27-2; **14a**, 75624-33-8; **14b**, 95070-28-3; **14c**, 26048-92-0; **14d**, 26191-98-0; **15a** (R = $PhCH_2$), 82933-25-3; **16a** (R = $PhCH_2$), 80543-45-9; 1-hydroxybenzotrizaole *syn*-2-(2-aminothiazol-4-yl)-2-(methoxyimino)acetate, 71445-20-0.

A Short, Stereocontrolled Synthesis of Avenaciolide

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The antifungal metabolite avenaciolide (1) is representative of a group of bislactone fungicides[1] which have

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