

from the crude mixture followed by recrystallization in EtOAc. As the overall route is concise and efficient, a practical scale-up process for terbinafine **1** is in progress.

In summary, terbinafine hydrochloride was prepared concisely *via* 4 steps in 27% overall yield.

Experimental

General. All commercial chemicals were used as obtained without further purification, and all solvents were carefully dried and distilled by standard methods prior to use. Column chromatography was carried out on silica gel 60 (E. Merck, 230-400 mesh) with the flash technique. Melting points were determined on a Thomas-Hoover melting point apparatus and uncorrected. Nuclear magnetic resonance spectra were determined on a Bruker ARX 300 spectrometer. Chemical shifts are reported in δ ppm relative to $(\text{CH}_3)_4\text{Si}$ for ^1H and ^{13}C NMR. Coupling constants, J are reported in Hz. Infrared spectra (cm^{-1}) were obtained on a Nicolet 710 FT-IR spectrometer. GC-mass analysis was performed on a Hewlett-Packard MSD 5890 series equipped with a capillary column (HP 1, 25 m).

6,6-dimethyl-2,4-diheptyn-1-ol (4). To a solution of 3-hydroxy-1-iodopropyne **3** (1.82 g, 10 mmol), *t*-Butyl acetylene **2** (1.5 mL, 12 mmol), $\text{Pd}(\text{PPh}_3)_4$ (350 mg), CuI (60 mg) in THF (40 mL) under a nitrogen atmosphere was added diisopropylamine (3.0 mL). After 3 hrs at room temperature the mixture was diluted with ether, washed with saturated ammonium chloride solution and brine, then dried over MgSO_4 . After concentration, the crude product was purified by column chromatography on silica gel (Hex : EtOAc = 5 : 1) to yield **4** (517 mg, 38%): ^1H NMR (CDCl_3) δ 4.32 (s, 2H), 1.85 (br, 1H), 1.24 (s, 9H); ^{13}C NMR (CDCl_3) δ 89.30, 75.01, 70.43, 63.04, 51.21, 30.21, 27.80; MS ($M^+ + 1$): 137.04; IR (film) ν 2970, 2252, 1022 cm^{-1} .

6,6-dimethyl-2E-hepten-4-yn-1-ol (5). To a solution of compound **4** (350 mg, 2.57 mmol) in THF (1.5 mL) was added sodium bis(2-methoxyethoxy)aluminum hydride (Red-Al from Aldrich, 65 wt% 1.27 g, 4.1 mmol) at 0 $^\circ\text{C}$. The reaction mixture was warmed to room temperature and stirred for 1 hr. The reaction mixture was quenched with 1.0 N sulfuric acid solution (1.5 mL), and the white solid was filtered off. Dilution with ether was followed by washing the organic phase with water twice and with brine once. The organic solution was dried over MgSO_4 . Evaporation of solvent under reduced pressure provided compound **5** quantitatively: ^1H NMR (CDCl_3) δ 6.20 (dt, J =15.9, 5.5, 1H), 5.84 (dt, J =15.9, 1.5, 1H), 4.21 (dd, J =5.5, 1.5, 2H), 1.64 (s, 1H), 1.31 (s, 9H); ^{13}C NMR (CDCl_3) δ 140.0, 112.0, 99.0, 63.0, 60.5, 31.0, 28.0; MS ($M^+ + \text{H}$): 139.04; IR (film) ν 2960, 2287, 1265 cm^{-1} .

Terbinafine (1), Terbinafine HCl salt. To a solution of compound **4** (300 mg, 2.17 mmol), Et_3N (1 mL), a catalytic amount of DMAP in methylene chloride (3.0 mL) was added methanesulfonyl chloride (0.336 mL, 4.34 mmol) at 0 $^\circ\text{C}$. After 5 mins. at 0 $^\circ\text{C}$, the reaction was quenched with water (2 mL) and stirred for 1 hr. Dilution with methylene chloride was followed by washing with water and drying over MgSO_4 . Evaporation of solvent on the rotatory evaporator afforded an oily compound. This crude product was added to a reaction mixture of *N*-methyl-1-naphthalenemethylamine (342.4

mg, 2.0 mmol), K_2CO_3 (1.5 g, 10.8 mmol) in DMF (1.5 mL). The reaction mixture was stirred for 6 hrs. at room temperature and then quenched with water. Extraction with EtOAc, washing with water, drying over MgSO_4 , and evaporation followed by separation by column chromatography afforded an oily compound **1**. For the more practical separation, the crude product was treated with HCl in methanol and evaporated to obtain a crude salt, and then the crude salt was treated with EtOAc to yield a white solid (448.1 mg, 77.0%): **terbinafine 1**; ^1H NMR (CDCl_3) δ 8.31 (m, 1H), 7.81 (m, 2H), 7.51 (m, 4H), 6.23 (dt, J =15.9, 6.6, 1H), 5.70 (dt, J =15.9, 1.4, 9H), 3.94 (s, 2H), 3.10 (dd, J =6.6, 1.4, 2H), 2.22 (s, 3H), 1.31 (s, 9H). Spectrum matches that reported in *J. Med. Chem.* **1984**, 27, 1539. **terbinafine hydrochloride**; mp 191-192 $^\circ\text{C}$ (lit.,^{3a} mp 195-198 $^\circ\text{C}$); ^1H NMR (DMSO) δ 10.9 (s, 1H), 8.4 (d, J =15.9, 1H), 8.1 (m, 2H), 7.9 (d, J =15.9, 1H), 7.6 (m, 3H), 6.2 (m, 1H), 6.0 (bd, 1H), 4.9 (m, 1H), 4.8 (m, 1H), 3.9 (m, 2H), 3.4 (s, 3H), 1.1 (s, 9H).

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Stereoselective Synthesis of (E)-Silyl Ketene Acetal from 2-Pyridyl Thioester¹

Kwee-Hyun Suh and Dong-Joon Choo*

Department of Chemistry, Kyung Hee University,
Seoul 130-701, Korea

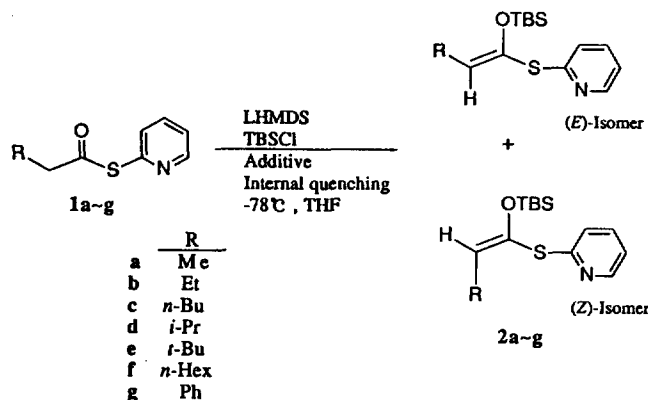
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Over the past decade, the stereoselective formation of ester and thioester enolates in aldol-type reactions has been the subject of intense investigation.² This is also the case for their related silyl ketene acetal,³ since Mukaiyama's disclosure⁴ for Lewis acid-mediated aldol condensation. During recent work⁵ on stereocontrolled Mukaiyama's aldol condensation, we have become interested in using the silyl ketene acetal of 2-pyridyl thioesters as aldol precursors.

Although aldol reaction with silyl ketene acetal of thioester is generally stereoconvergent regardless of the geometry of silyl ketene acetal,^{3,6} we needed the stereoselective synthesis

of silyl ketene acetal of 2-pyridyl thioester in order to understand the stereochemical outcome of the subsequent aldol reaction. Here we report that stereoselective formation⁷ of (*E*)-silyl ketene acetal from 2-pyridyl thioester can be achieved by proper selection of the reaction conditions as described in Scheme 1. The level of stereoselectivity thus obtained could be explained by the thermodynamically controlled model for enolate generation.

The starting 2-pyridyl thioesters **1** were prepared from the corresponding acid chloride and 2-mercaptopyridine according to known methods.⁸ As a preliminary study, we tried to synthesize the silyl ketene acetal **2a** from 2-pyridyl thiopropionate **1a**. But the preparation of 2-pyridylthio silyl ketene acetal using conventional enolization method⁹ was not successful, probably due to the inherent tendency of 2-pyridylthio moiety acting as a leaving group. It has been discovered that this problem could be circumvented by internal quenching¹⁰ of lithium enolate of **1a** with silyl agent, thereby the silyl ketene acetal **2a** was efficiently synthesized from 2-pyridyl thiopropionate. Thus 2-pyridyl thiopropionate was added to a mixture of one equivalent of lithium 1,1,1,3,3,3-hexamethyldisilylamide (LHMDS) and one equivalent of *tert*-



Scheme 1.

butyl-dimethylsilyl chloride (TBSCl) in the presence of 2-4 equivalents of additives such as hexamethylphosphoramide (HMPA), *N,N'*-dimethyl-propyleneurea (DMPU), 1,1,3,3'-tetramethylurea (TMU), or *N,N*-dimethylformamide (DMF) at -78°C in THF. After usual aqueous work up and purifica-

Table 1. The stereoselective synthesis of *tert*-butyldimethylsilyl ketene acetal **2** from 2-pyridyl thioester **1**^a

Entry	R	Base ^b	Type ^c	Additive (eq.) ^d	Yield, % ^d	E/Z ^e
1	Me	LDA	N	none	<5(NMR)	
2	Me	LDA	R	none	9(NMR)	
3	Me	LDA	N	HMPA(4.5)	trace(NMR)	
4	Me	LHMDS	N	none	28	82/18
5	Me	LHMDS	R	none	26	84/16
6	Me	LHMDS	R	TEA(2.0)	27	85/15
7	Me	LHMDS	R	TEA(4.0)	25	90/10
8	Me	LHMDS	R	LiCl(1.0)	40	85/15
9	Me	LHMDS	R	TMEDA(1.0)	trace(NMR)	
10	Me	LHMDS	R	HMPA(2.0)	58	>96/4
11	Me	LHMDS	R	HMPA(4.0)	56	>98/2
12	Me	LHMDS	R	DMPU(2.0)	79	>98/2
13	Me	LHMDS	R	DMPU(4.0)	80	>99/1
14	Me	LHMDS	R	TMU(2.0)	61	>96/4
15	Me	LHMDS	R	TMU(4.0)	65	>98/2
16	Me	LHMDS	R	DMF(2.0)	73	>96/4
17	Me	LHMDS	R	DMF(4.0)	78(86) ^f	>98/2
18	Et	LHMDS	R	DMF(4.0)	72	>99/1
19	n-Bu	LHMDS	R	DMF(4.0)	75	>99/1
20	i-Pr	LHMDS	R	DMF(4.0)	54	>99/1
21	t-Bu	LHMDS	R	DMF(4.0)	trace(NMR)	
22	n-Hex	LHMDS	R	DMF(4.0)	73	>99/1
23	Ph	LHMDS	R	DMF(4.0)	70	82/20
24	Me	LDA	R	DMF(4.0)	trace(NMR)	
25	Me	LDCA	R	DMF(4.0)	trace(NMR)	
26	Me	LTMP	R	DMF(4.0)	trace(NMR)	
27 ^g	Me	LHMDS	R	DMF(4.0)	12	>98/2
28	^h	LHMDS	R	DMF(4.0)	85	>99/1

^aThe reactions were carried out in 1-3 mmole scale. ^bThe full names for abbreviations were given in manuscript. ^cAddition type of TBSCl, N: normal addition, R: reverse addition (internal quenching). ^dIsolated yield after chromatography. ^eBased on 200 MHz ¹H NMR spectra of crude product. ^fYield in 15 mmole scale was in parenthesis. ^gTMSCl instead of TBSCl was employed. ^hS-Phenyl thiopropionate was used.

tion by flash chromatography, the (*E*)-silyl ketene acetal **2a** was obtained in high stereoselectivity and good yield. The results are summarized in Table 1.

It is clear from Table 1 that initial trials using lithium diisopropylamide (LDA) for the generation of lithium enolate from 2-pyridyl thiopropionate did not give a substantial amount of silyl ketene acetal **2a**, regardless of the type of addition of trapping agent, TBSCl, as shown in Entries 1 and 2. Also, Ireland's method (Entry 3)¹¹ using LDA and HMPA (4.5 eq.) in THF was not effective. However the use of LHMDS as a base afforded a mixture of (*E*)- and (*Z*)-silyl ketene acetal **2a** in 82/18 (normal addition) and 84/16 (reverse addition) ratio according to addition type of TBSCl, and yields were slightly increased as represented in Entries 4 and 5 of Table 1. The addition of triethylamine (2-4 eq., Entries 6 and 7) or lithium chloride¹² (1 eq., Entry 8) to reaction mixture displayed slight increase of the (*E*)-selectivity and yield respectively, but the use of *N,N,N',N'*-tetramethylethylenediamine¹³ (TMEDA, Entry 9) led to severe decomposition of thioester **1a** in enolization step.

The most satisfactory results in the formation of the (*E*)-silyl ketene acetal **2a** from thioester **1a** were obtained by the use of LHMDS as a base and highly polar additives such as HMPA, DMPU, TMU or DMF in reverse addition of TBSCl as a silylating agent as shown in Entries 10-17 of Table I. Under this optimal condition employing 2-4 equivalents of these additives, the (*E*)-silyl ketene acetal of 2-pyridyl thioester was obtained in high stereoselectivity (*E*/*Z* = > 96/4, in all cases) and good yield. The ratios of (*E*)- and (*Z*)-isomers were determined by 200 MHz ¹H NMR spectroscopy of crude product, and the geometry of major product was assigned to (*E*)-configuration by observing the occurrence of NOE between protons of allylic methyl group and dimethyl protons on silicon of **2a** from 600 MHz ¹H NMR. Moreover, no NOE between α -vinyl proton and SiMe₂ (*tert*-Bu) group was detected in our NOE spectroscopic experiment.

The extension of the present method to other 2-pyridyl thioesters such as **1b** (R=ethyl), **1c** (R=*n*-butyl), **1d** (R=*i*-propyl), **1e** (R=*tert*-butyl), **1f** (R=*n*-hexyl), and **1g** (R=phenyl) also showed the high (*E*)-selectivity and good yield except **1e**, which possesses bulky substituent in α -position, as listed in Entries 18-23 of Table 1. The high level of the (*E*)-selectivity in silyl ketene acetal formation from 2-pyridyl thioester **1** is comparable to that of Ireland's,¹¹ Gennari's,⁶ or Otera's¹⁴ cases for ethyl propionate, *tert*-butyl thiopropionate, and *tert*-butyl propionate, respectively. This *E* selectivity could be basically explained in terms of Ireland's postulate¹¹ of cyclic transition states **Z** and **E** for enolization as represented in Figure 1. Although there are various experimental evidences¹⁵ which suggest the involvement of aggregated species in enolization, the degree of the solvation between the employed additives (or solvents), lithium cation of the amide base is still the crucial factor in the interpretation of steric requirement for enolization in Ireland's model.

As shown in Figure 1, the stronger solvation of the lithium amide in the presence of highly polar additives such as HMPA, DMPU, TMU, and DMF would surely enhance the reactivity of the amide base, thus the lithium-carbonyl oxygen interaction in the transition state **E** leading (*E*)-silyl ketene acetal should be much weaker than in transition state **Z** leading (*Z*)-isomer. In that situation, the nonbonded inter-

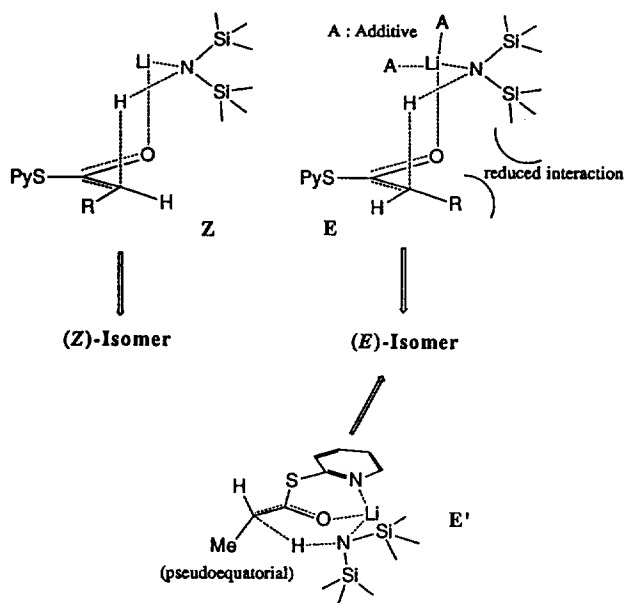


Figure 1. The proposed Model for (*E*)-Selectivity.

action between R group of thioester and the amide base would not be effective. Accordingly, R could become eclipsed with the smaller carbonyl oxygen and positioned *trans* to Py-S- group as in transition state **E**. However, high (*E*)-selectivity was obtained even in the absence of additive as shown in Entries 4 and 5, albeit in low yield. This (*E*)-selectivity cannot be deduced by Ireland's model. Instead, it would be expected that the boat-like cyclic transition state **E'** (Figure 1) should be the more appropriate model, in which pyridine nitrogen of thioester **1a** would participate in enolization step and methyl group lie in pseudo-equatorial position. It should also be pointed out that the lowered selectivity (*E*/*Z* = 80/20) for (*E*)-isomer in the case of **2g** (Entry 23), which possesses -protons with enhanced acidity,¹⁶ cannot be explained in terms of these cyclic transition states for the steric or stereoelectronic requirement.

All other trials (Entries 18, 19, and 26) using stronger base such as LDA, lithium dicyclohexylamide (LDCA), or lithium 1,1,6,6-tetramethylpiperidine (LTMP) than LHMDS were not successful. The ¹H NMR spectra of their reaction mixtures showed only trace amounts of silyl ketene acetal **2a**. The use of trimethylsilyl chloride (TMSCl, Entry 27) instead of TBSCl as a trapping silyl agent retained high stereoselectivity for (*E*)-isomer but yield was very low. This is probably due to the sensitivity of trimethylsilyl ketene acetal to water during aqueous work up. In the case of phenyl thiopropionate in which the 2-pyridyl group of **1a** is replaced by a phenyl group (Entry 28), the excellent stereoselectivity for (*E*)-silyl ketene acetal and high yield were observed. From these observations, it would be safe to conclude that the present method could be applicable to the stereoselective synthesis of (*E*)-silyl ketene acetals from various thioesters as well as 2-pyridyl thioester **1**. Further stereoselective synthesis of (*Z*)-silyl ketene acetal from 2-pyridyl thioester and the subsequent aldol condensation with the (*E*)- and (*Z*)-silyl ketene acetals are under investigation.

Experimental Section

General Procedure. for the synthesis of (*E*)-silyl ketene acetal **2** from 2-pyridyl thioester **1**: A solution of 462 μ L (2.2 mmol) of 1,1,1,3,3,3-hexamethyldisilazane in 14 mL of dried THF cooled to -15°C under nitrogen, and 1.4 mL of a 1.6 M solution of *n*-BuLi in hexane was added slowly by syringe. This mixture was stirred for 10 min at -10°C and cooled to -78°C , and 2-4 equivalents of additives was added dropwise by syringe. After 10 min, a solution of 365 mg (2.2 mmol) of TBSCl in 3 mL of THF was added by syringe. After an additional 3 min, a solution of 2.0 mmol of thioester **1** in 3 mL of THF was added dropwise by syringe under intense stirring. The resulting solution (or suspension) was stirred for 30 min at -78°C , allowed to warm up to 0°C over 30 min, and quenched with water. The mixture was extracted with ethyl ether three times. The combined organic phase was washed with water, dried over MgSO_4 , and concentrated to leave yellow liquid. After the isomer ratio was determined by ^1H NMR spectroscopy, the crude product was purified by flash chromatography (silica gel, hexane/ether=90/10) to give the (*E*)-silyl ketene acetal **2** as colorless oil. 200 MHz ^1H NMR spectroscopic data of the (*E*)-**2** in CDCl_3 are as the follows;

(*E*)-**2a**: δ 0.10 (s, 6H), 0.89 (s, 9H), 1.74 (d, $J=6.7$ Hz, 3H), 5.46 (q, $J=6.7$ Hz, 1H), 6.98-7.03 (dd, 1H), 7.32 (d, $J=8.6$ Hz, 1H), 7.50-7.61 (m, 1H), 8.42 (d, $J=4$ Hz, 1H).

(*E*)-**2b**: δ 0.09 (s, 6H), 0.89 (s, 9H), 1.04 (t, $J=7.4$ Hz, 3H), 2.21 (quintet, $J=7.4$ Hz, 2H), 5.41 (t, $J=7.2$ Hz, 1H), 6.98-7.03 (dd, 1H), 7.32 (d, $J=8.6$ Hz, 1H), 7.52-7.62 (m, 1H), 8.41 (d, $J=4$ Hz, 1H).

(*E*)-**2c**: δ 0.09 (s, 6H), 0.79-1.01 (m, 12H), 1.22-1.49 (m, 4H), 2.20 (m, 2H), 5.42 (t, $J=7.32$ Hz, 1H), 6.98-7.03 (dd, 1H), 7.32 (d, $J=8.6$ Hz, 1H), 7.52-7.62 (m, 1H), 8.41 (d, $J=4.0$ Hz, 1H).

(*E*)-**2d**: δ 0.10 (s, 6H), 0.90 (s, 9H), 1.05 (d, $J=6.8$ Hz, 6H), 2.81 (m, 1H), 5.23 (d, $J=9.2$ Hz, 1H), 6.98-7.03 (dd, 1H), 7.32 (d, $J=8.6$ Hz, 1H), 7.52-7.62 (m, 1H), 8.41 (d, $J=4.0$, 1H).

(*E*)-**2f**: δ 0.09 (s, 6H), 0.83-1.03 (m, 12H), 1.20-1.51 (m, 8H), 2.21 (dt, 2H), 5.43 (t, $J=7.3$ Hz, 1H), 6.98-7.03 (dd, 1H), 7.32 (d, $J=8.6$ Hz, 1H), 7.52-7.62 (m, 1H), 8.41 (d, $J=4.0$ Hz, 1H).

(*E*)-**2g**: δ 0.11 (s, 6H), 0.90 (s, 9H), 6.33 (s, 1H), 7.01-7.70 (m, 8H), 8.46-8.51 (m, 1H).

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