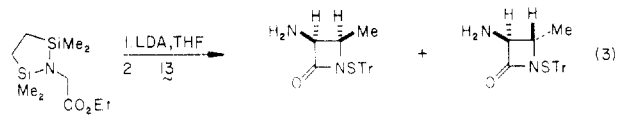
5 and 13 is one of the few examples of β-lactam formation in a reaction involving lithium enolates and an enolizable azomethine.[13-15] Finally, the reactions show stereoselectivities which parallel results obtained with simple lithium enolates and N-trimethylsilyl or N-arylaldimines.[2]

The potential of this route to β-lactams is underscored by the example shown in eq 3. Thus, treatment of glycine



15 16 17

derivative **15**[16] with sulfenimine **13** (−20 °C, 4 h) gave a 5:1 mixture of **16** (mp 146–147.5 °C) and **17**, respectively, in a 78% yield after chromatography over silica gel. The relationship of this reaction to monobactam synthesis is obvious.[17,18]

18 19

In keeping with the objective of developing procedures for the preparation of N-unsubstituted β-lactams, several methods for reductive removal of the N-tritylsulfenyl group were examined. It was found that this task could be accomplished by using several reagents. For example, treatment of β-lactam **11** with tri-*n*-butylphosphine (10 equiv, EtOH–THF, 115 °C, 48 h) gave **3** (75%). Treatment of **11** with trimethylsilyl iodide (CH_2Cl_2, 25 °C, 7 h) also gave **3** (81%).[19] Although 4-alkylated β-lactam **14a** could be converted to **9** by using freshly prepared W-2 Raney nickel[20] (40%) or lithium in ammonia (85%), these conditions resulted in the conversion of 4-arylated β-lactam **11** to amide **18** in 45% and 79% yields, respectively.[21,22] Finally, when **11** was warmed with anhydrous $CuCl_2$ (2 equiv) in tetrahydrofuran–ethanol (75 °C, 5 h), only S–C bond cleavage was observed.[23] Thus, disulfide **19** was obtained in 69% yield as a mixture of diastereomers. This reaction may be particularly useful in monobactam synthesis.

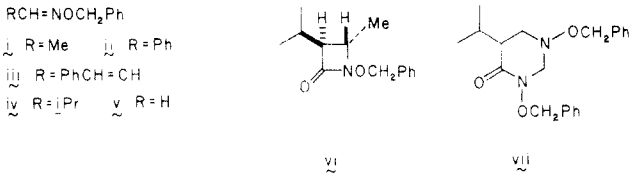
In summary, the condensation of lithium ester enolates with sulfenimines represents a promising new entry to β-lactams. Additional studies including applications to monobactam and carbapenem total synthesis as well as ketene–sulfenimine cycloadditions are underway.[24]

Acknowledgment. We thank Mr. Richard Weisenberger for recording mass spectra at The Ohio State University Campus Chemical Instrument Center. Financial support from the National Institutes of Health (AI-21074) is gratefully acknowledged.

Registry No. 1, 97-62-1; **2a**, 52777-99-8; **2b**, 61501-00-6; **3**, 7486-93-3; **4**, 51364-95-5; **5**, 108-64-5; **6**, 90696-11-0; **7**, 101518-51-8; **8**, 101518-52-9; **9**, 101518-53-0; **10**, 86864-34-8; **11**, 101518-54-1; **12a**, 101518-55-2; **12b**, 101541-77-9; **13**, 101518-56-3; **14a**, 101518-56-3; **14b**, 101518-58-5; **15**, 78605-23-9; **16**, 101518-59-6; **17**, 101518-60-9; **18**, 101518-61-0; **19**, 101518-62-1; **i**, 20134-98-9; **v**, 72399-18-9; **vi**, 101518-63-2; **vii**, 101518-64-3; ethyl 2-bromoisovalerate, 609-12-1.

Supplementary Material Available: Procedure for preparation of **13** (1 page). Ordering information is given on any current masthead page.

(12) Our own studies have shown that the lithium enolate of **5** in THF gives no β-lactam with oxime ethers i and ii. Only the product of apparent Claisen condensation of **5** is obtained (50%). The enolate derived from sequential treatment of **5** (1 equiv) with LDA (1 equiv) and $ZnCl_2$ (1 equiv) in THF gives only trace amounts of β-lactams upon treatment with oxime ethers ii–iv. This enolate, however, does give a 27% yield of vi and a 45% yield of vii upon treatment with oxime ethers i and v, respectively. Treatment of ethyl 2-bromoisovalerate with Zn in THF and i also gives vi (21%).



vi vii

(13) Although not widely used, enolizable *N*-aryl- and *N*-alkylaldimines react with zinc enolates (Reformatsky reagents) to give β-lactams in certain situations: Dardoize, F.; Moreau, J.-L. Gaudemar, M. *C. R. Hebd. Seances Acad. Sci., Ser. C* **1969**, *268*, 2228. Dardoize, F.; Moreau, J.-L.; Gandemar, M. *Bull. Soc. Chim. Fr.* **1972**, 3841. Lithium enolates have been reported to give no β-lactam upon treatment with enolizable *N*-alkylaldimines: Gluchowski C.; Cooper, L.; Bergbreiter, D. E.; Newcomb, M. *J. Org. Chem.* **1980**, *45*, 3413.

(14) For β-amino ester formation from a lithium enolate and an enolizable *N*-(tetrazol-5-yl)aldimine, see: Klich, M.; Teutsch, G. *Tetrahedron Lett.* **1984**, *25*, 3849.

(15) Acid-catalyzed reactions of enolizable imines and ester enolates to afford β-amino esters are known (Volkmann, R. A.; Davis, J. T.; Meltz, C. N. *J. Am. Chem. Soc.* **1983**, *105*, 5946.) as are reactions between (vinyloxy)boranes and enolizable imines: Otsuka, M.; Yoshida, M.; Kobayaski, S.; Ohno, M.; Umezawa, Y.; Morishima, H. *Tetrahedron Lett.* **1981**, *22*, 2109. Iimori, T.; Shibasaki, M. *Tetrahedron Lett.* **1985**, *26*, 1523.

(16) Djuric, S.; Venit, J.; Magnus, P. *Tetrahedron Lett.* **1981**, *22*, 1787.

(17) Floyd, D. M.; Fritz, A. W.; Cimarusti, C. M. *J. Org. Chem.* **1982**, *47*, 176. Koster, W. H.; Cimarusti, C. M.; Sykes, R. B. In *Chemistry and Biology of β-Lactam Antibiotics*; Morin, R. B., Goldman, M., Eds.; Academic Press: New York, 1982; Vol. 3, pp 339–375.

(18) Reference 14 describes a related but lengthier approach to *N*-(tetrazol-5-yl)-2-azetidinones.

(19) Trimethylsilyl iodide has previously been used to cleave N–S bonds of *S*-tritylsulfenamides: Branchaud, B. P. *J. Org. Chem.* **1983**, *48*, 3538.

(20) Mozingo, R. *Organic Syntheses*; Wiley: New York; 1955; Collect. Vol. 3, p 181.

(21) If freshly prepared W-2 Raney nickel was warmed at 75 °C in ethanol for 48 h followed by treatment with **11** under 1 atm of hydrogen at 25 °C, β-lactam **3** (79%) was obtained.

(22) The generality of these N–S to N–H transformations remains to be established. Details will be reported in our full account of this research.

(23) These conditions have been used to cleave N–S bonds of *S*-tritylsulfenimides.[19]

(24) **Note Added in Proof**: We have shown that the lithium enolate of **1** reacts with the *N*-tritylsulfenyl imine of acetone to give 3,3,4,4-tetramethyl-1-(tritylsulfenyl)-2-azetidinone in 60% yield.

(25) Phillips Petroleum Graduate Fellow, 1985.

(26) Fellow of the Alfred P. Sloan Foundation, 1983–1987.

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The ((β-Phenylethyl)oxy)carbonyl ("Homobenzyloxycarbonyl", hZ) Amino-Protecting Group

Summary: The ((β-phenylethyl)oxy)carbonyl group ("homobenzyloxycarbonyl", hZ) is shown to be generally deblocked by catalytic hydrogenolysis, especially via the catalytic transfer technique using freshly precipitated palladium–carbon in the presence of ammonium formate.

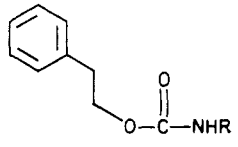
0022-3263/86/1951-1930$01.50/0

Table I. Preparation and Deblocking of β-Phenylethyl-Substituted Urethanes and Esters

entry	compound[a]	mp (°C), yield (%)	conditions[b]	result[c] (%)
1	hZ-Gly-OBn	54 (90)	S.C.	H-Gly-OH (100)
2	hZ-Gly-OBn	54 (90)	H_2/Pd–C/25 °C/8 h[d]	hZ-Gly-OH (95)
3	hZ-Gly-Gly-O$(CH_2)_2C_6H_5$	115 (68)	S.C./65 °C/15 h	H-Gly-Gly-OH (100)[e]
4	hZ-Phe-Leu-O-*t*-Bu	*f*	S.C./65 °C/8 h[g]	H-Phe-Leu-O-*t*-Bu (100)
5	hZ-Gly-Gly-Phe-Leu-O-*t*-Bu	*h*	S.C./sonication/50 °C/4 h	H-Gly-Gly-Phe-Leu-O-*t*-Bu (100)
6	$C_6H_5COO(CH_2)_2C_6H_5$	*i*	S.C./65 °C/24 h	$C_6H_5CO_2H$ (90)[j]
7	$C_6H_5O(CH_2)_2C_6H_5$	*k*	S.C./65 °C/24 h	no deblocking observed
8	$C_6H_5NH(CH_2)_2C_6H_5$	*l*	S.C./65 °C/15 h	no deblocking observed
9	hZ-NHC_6H_5	*m*	S.C.	$C_6H_5NH_2$ (90)[n]
10	hZ-NHC_6H_5	*m*	HBr/HOAc/24 h	recovery of urethane (92)
11	BOC-Gly-O$(CH_2)_2C_6H_5$	bp 150 (0.8 mm) (64)[o]	HCl/Et_2O/5 min	HCl·H-Gly-O$(CH_2)_2C_6H_5$ (100)
12	Bn | hZ-Tyr-Gly-Gly-Phe-Leu-O-*t*-Bu	*p*	CF_3CO_2H/CH_2Cl_2/25 °C/24 h	Bn | hZ-Tyr-Gly-Gly-Phe-Leu-OH(94)

[a] Obtained via acylation with β-phenylethyl chloroformate or as indicated. [b] Standard conditions (S.C.) involved 100 mg of each of the following: 10% Pd–C, $Pd(OAc)_2$, and substrate along with 300 mg of NH_4OCHO in 10 mL of MeOH at 25 °C for 8 h. Variations are recorded. Under sonication, reactions were performed in an ordinary ultrasonic laboratory cleaner (Branson B-12) with spontaneous increase in the temperature to 50 °C. The byproduct, ethylbenzene, is not normally observed due to its volatility although in several cases its presence was demonstrated by NMR analysis. [c] The identity of all compounds was established by comparison with authentic IR and ^1H NMR spectra. [d] Reaction performed by passing H_2 through the solution at 1 atm. [e] At 25 °C partial deblocking occurred to give mainly H-Gly-Gly-O$(CH_2)_2C_6H_5$. [f] From hZ-Phe-OH [mp 48 °C 91%, α^{20}_D +48.25° (*c* 0.4, $CHCl_3$)] by conversion to the acid chloride via $SOCl_2$ (2 h, reflux, CH_2Cl_2) followed by reaction with H-Leu-O-*t*-Bu/H_2O/$NaHCO_3$/CH_2Cl_2: yield 41%; mp 118 °C; α^{20}_D −6.02° (*c* 1.42, $CHCl_3$). Model studies showed no significant racemization (<0.1% by HPLC analysis). [g] The corresponding methyl ester was deblocked completely at 25 °C over a period of 20 h. [h] Obtained via hZ-Gly-Gly-Cl and crude deblocked product from entry 4 in $H_2O/CH_2Cl_2/NaHCO_3$: yield 75%; mp 129 °C, α^{20}_D −19.58° (*c* 0.965, MeOH). [i] Golovina, Z. P.; Bogatkov, S. V.; Cherkasova, E. M. *J. Org. Chem. (USSR)* **1975**, *11*, 20. [j] Obtained via acidification of the ammonium salt which was isolated directly from the reaction mixture upon filtration to remove catalyst and evaporation of solvent and excess NH_4OCHO. It should be noted that the palladium catalyst effects decomposition of NH_4OCHO into H_2, CO_2, and NH_3, the solution rapidly becoming basic. [k] Ermokhina, V. A.; Arifova, D. U. *Sb. Nauch. Tr. Tashkent. Un-t.* **1976**, 48 [*Chem. Abstr.* **1978**, *89*, 179630]. [l] Malkhasyan, A. Ts.; Dzhandzhulyan, Zh. L.; Martirosyan, G. T. *Arm. Khim. Zh.* **1978**, *31*, 870 [*Chem. Abstr.* **1979**, *90*, 137374]. [m] Walbaum, H. *Ber.* **1900**, *33*, 2299. [n] Isolated as acetanilide by reaction with AcCl and NEt_3. [o] Glycine β-phenylethyl ester *p*-toluenesulfonate was prepared by adaptation of a method recommended for the corresponding benzyl ester [Zervas, L; Winitz, M.; Greenstein, J. P. *J. Org. Chem.* **1957**, *22*, 1515]. Twenty-hour treatment with N_3COO-*t*-Bu/NEt_3 then gave the protected ester. [p] Obtained via hZ-Tyr(Bn)-OH [mp 92 °C, α^{20}_D +74.52 (*c* 0.84, $CHCl_3$), 54%] and the crude product from entry 5 by DCC coupling: yield 85%, mp 153 °C, structure confirmed by 300-MHz ^1H–^1H 2D chemical shift correlation spectroscopy (HOMCOR) [Bax, A.; Freeman, R. *J. Magn. Reson.* **1981**, *44*, 542]. Further deblocking of the free acid under sonication gave leucine enkephalin, identified by amino acid analysis, and comparison (IR, NMR,[q] TLC, HPLC) with an authentic sample (Chemical Dynamics). [q] Garbay-Jaureguiberry, C. J.; Roques, B. P.; Oberlin, R.; Anteunis, M.; Combrisson, S.; Lallemand, J. Y. *FEBS Lett.* **1977**, *76*, 93.

Sir: Modern peptide synthesis began with the observation of Bergmann and Zervas[1] that the benzyloxycarbonyl ("carbobenzoxy", Z) group is readily cleaved under mild conditions by catalytic hydrogenolysis with the generation of the innocuous byproducts carbon dioxide and toluene. We now call attention to the fact that the ((β-phenylethyl)oxy)carbonyl group ("homobenzyloxycarbonyl" or "homocarbobenzoxy", hZ) as in urethane 1 is also subject



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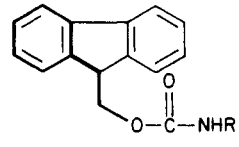

2

to reductive catalytic deblocking, although at a somewhat reduced rate. Discovery of the general sensitivity of 1 to hydrogenolysis followed our belated recognition[2] of the surprising susceptibility to reduction of the ((9-fluorenylmethyl)oxy)carbonyl protecting function, as in 2, in which is embedded the β-phenylethyl structural element. Deblocking was generally carried out by the catalytic transfer hydrogenolytic method using ammonium formate in methanol.[7] The most active system involved a freshly prepared catalyst.[8] Rate differences between benzyl- and β-phenylethyl-based systems can be great enough to allow selective cleavage. As an example, the *N*-homobenzyloxycarbonyl derivative of benzyl glycinate[9] was found to be deblocked only at the benzyl ester group at atmosheric pressure over palladium–carbon catalyst (10%) (Table I, entry 2) whereas catalytic transfer hydrogenolysis by means of ammonium formate removed both arylalkyl functions (entry 1).

Where a protecting group must survive strongly acidic conditions, the use of benzyl-based systems may be contraindicated[10] and β-phenylethyl-derived reagents could

(1) Bergmann, M.; Zervas, L. *Chem. Ber.* **1932**, *65*, 1192.

(2) In our first description[3] of the utility of the FMOC amino-protecting group, we reported complete stability of this function toward catalytic hydrogenolysis over palladium catalysts. Subsequently, Bodanszky[4] and Sheppard[5] and their co-workers observed the deblocking of FMOC derivatives over similar catalysts. Other investigators[6] confirmed our original observations and carried out clean catalytic debenzylations in the presence of FMOC protection. These confusing results appear to be due to the activity of the various catalysts used. Our original studies were carried out with catalysts that had been deactivated by long standing. Freshly prepared catalysts ensure the fastest deblocking with FMOC systems. Still, with whatever catalysts are chosen the FMOC group is cleaved at substantially lower rates than benzyl-based functions. As observed in the present work, there is a similar difference between the benzyloxycarbonyl and the homobenzyloxycarbonyl systems. Although no quantitative data are available, for the three urethanes the susceptibility toward hydrogenolysis appears to follow the order hZ < FMOC < Z.

(3) Carpino, L. A.; Han, G. Y. *J. Am. Chem. Soc.* **1970**, *92*, 5748; *J. Org. Chem.* **1972**, *37*, 3404.

(4) Martinez, J.; Tolle, J. C.; Bodanszky, M. *J. Org. Chem.* **1979**, *44*, 3596.

(5) Atherton, E.; Bury, C.; Sheppard, R. C.; Williams, B. J. *Tetrahedron Lett.* **1979**, 3041.

(6) (a) Chang, C.-D.; Waki, M.; Ahamd, M.; Meienhofer, J.; Lundell, E. O.; Haug, J. D. *Int. J. Pept. Prot. Res.* **1980**, *15*, 59. (b) Almquist, R. G.; Christie, P. H.; Chao, W.-R.; Johnson, H. L. *J. Pharm. Sci.* **1983**, *72*, 63.

(7) Anwer, M. K.; Spatola, A. F. *Synthesis* **1980**, 929.

(8) Best results were obtained with palladium freshly precipitated from the acetate onto commercial 5–10% palladium–carbon by reduction with ammonium formate.

(9) All new compounds gave correct C, H, N values (±0.3%) and appropriate IR and NMR spectral data.

be advantageous. Thus, although a standard technique for removal of the benzyloxycarbonyl group involves treatment with hydrogen bromide in acetic acid,[11] the homobenzyloxycarbonyl function is stable in this reagent over a period of 24 h at room temperature. Hydrogen chloride in ether readily cleaves the *t*-BOC group of entry 11 (Table I)[9] or trifluoroacetic acid the *tert*-butyl ester function of the protected pentapeptide of entry 12 without affecting the β-phenylethyl ester or hZ functions, respectively. The stability of the hZ group toward acids is further demonstrated by the conversion of protected amino acids to the corresponding acyl chlorides which can be used as coupling agents (Table I, entries 4 and 5).

In cases where the catalytic deblocking of hZ functions proceeded slowly, reaction could be speeded up by increasing the ratio of catalyst to substrate, sonication,[12] and/or heating to 50–65 °C. When incorporated in ester form the β-phenylethyl residue is cleaved less readily than the corresponding urethane structure. Evidence for the relative stability of β-phenylethyl glycinate toward catalytic hydrogenolysis has been cited previously.[13] Steric or other factors may influence the deblocking process. Thus, in the case of the methyl ester related to the dipeptide derivative of entry 4 (Table I), catalytic transfer hydrogenolysis removed the hZ group within 20 h at room temperature whereas the *tert*-butyl ester survived reaction under the same conditions.

In view of the ready availability[14] and shelf stability of β-phenylethyl chloroformate, the homobenzyloxycarbonyl amino-protecting group promises to be of significant potential applicability in organic synthesis. Synthesis of hZ derivatives of amino acids follows standard procedures. The derivative of glycine has been reported in another connection.[15]

Preparation of Catalyst. To 10 mL of ice-cold methanol was added 100 mg of 5 or 10% Pd–C catalyst (Pfaltz and Bauer or Fluka). The mixture was stirred magnetically and treated immediately with 100 mg of palladium acetate and 300 mg of ammonium formate. After about 1 min a flocky suspension of the catalyst was formed as the color of the palladium salt disappeared. The substrate to be deblocked (100–500 mg)[16] was added and the mixture stirred at room temperature until the starting material was consumed (TLC, HPLC). Filtration to remove the catalyst was followed by evaporation of ammonia, methanol, and volatile NH_4OCHO at atmospheric or reduced pressure. The residue was purified in an appropriate manner. For examples see the table.

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(10) (a) Noda, K.; Terada, S.; Izumiya, N. *Bull. Chem. Soc. Jpn.* **1970**, *43*, 1883. (b) Yamashiro, D.; Li, C. H. *J. Am. Chem. Soc.* **1973**, *95*, 1310. (c) Blaha, K.; Rudinger, J. *Collect. Czech. Chem. Commun.* **1965**, *30*, 585. For studies on the relative stability of β-phenylethyl esters toward acidic conditions, see: Tronow, B. W.; Ssibgatullin, N. C. *Chem. Ber.* **1929**, *62*, 2850.

(11) Ben-Ishai, D.; Berger, A. *J. Org. Chem.* **1952**, *17*, 1564.

(12) Ultrasound has previously been used to enhance the rate of catalytic hydrogenation processes. For examples and a general discussion, see: (a) Boudjouk, P. *Nachr. Chem. Tech. Lab.* **1983**, *31*, 798. (b) Boudjouk, P.; Han, B.-H. *J. Catal.* **1983**, *79*, 489. (c) Han, B.-H.; Boudjouk, P. *Organometallics* **1983**, *2*, 769.

(13) Taylor-Papadimitriou, J.; Yovanidis, C.; Paganou, A.; Zervas, L. *J. Chem. Soc. C* **1967**, 1830. See, however, entries 3 and 6 of the table.

(14) Najer, H.; Chabrier, P.; Giudicelli, R. *Bull. Soc. Chim. Fr.* **1955**, 1189.

(15) Barltrop, J. A.; Schofield, P. *J. Chem. Soc.* **1965**, 4758.

(16) In order to maintain a reasonable rate of hydrogenolysis for reactions run at room temperature, a greater weight of active palladium is generally required over that needed for comparable benzylic systems. For deblockings run at or somewhat below the reflux temperature of methanol, or under sonication, less catalyst may be used. Under reflux in methanol, ammonium formate collects in the condenser and should be pushed back into the reaction mixture occasionally. For some substrates optimal results require freshly precipitated palladium whereas in other cases an additional quantity of the commercial palladium–carbon catalyst is sufficient. More active as well as more selective catalysts are being sought.

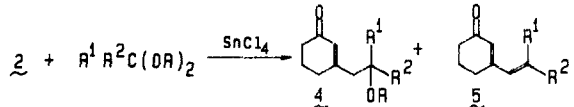
Reactions of 3-[(Trimethylsilyl)methyl]cyclo-2-hexenone with Carbonyl Compounds. Regio- and Chemoselective Condensations

Summary: Under the influence of $SnCl_4$ or trimethylsilyl iodide the title compound reacts with acetals or aldehydes at the γ-position to give the corresponding condensation products, whereas fluoride or base-mediated reactions take place at the α-position to afford α-substituted products selectively.

Sir: Structures of cyclohexenones bearing unsaturated side chains at their 3-position are common carbon frameworks in a wide range of natural products such as vitamin A, retinal, and so on. It appears that such skeletons could be constructed from the corresponding enol silyl ether such as **1**. Enol silyl ethers of unsaturated carbonyl compounds have been used in a wide range of synthetic organic chemistry and a variety of procedures have been developed for the regioselective synthesis of such substrates,[1,2a] but we still could not find any reliable method for the preparation of compounds similar to **1**.[2]

Judging from the mode of reactivities of α-silyl ketones,[3] the keto isomer **2** is expected to behave in a similar manner with **1** in Lewis acid mediated reactions with carbonyl compounds or their acetals. In this paper, we describe a preparative method for keto isomer **2** and its synthetic use for carbon-chain homologation. The silyl ketone **2** can be prepared as follows. Treatment of cyclohexenone with [(trimethylsilyl)methyl]magnesium chloride in the presence of a catalytic amount of CuBr–SMe_2 and chlorotrimethylsilane (1.1 equiv)[4] led to the direct formation of the corresponding enol silyl ether **3** of 3-[(trimethylsilyl)methyl]cyclohexanone. The resulting **3** was converted to the desired **2** by the reaction with $^1/_4$ equiv of palladium acetate and allyl carbonate[5] in good overall yield (Scheme I).

Under the influence of $SnCl_4$, the silyl ketone **2** reacts with various acetals (eq 1). Interestingly, the reaction



(1)

(1) (a) Colvin, E. W. *Silicon in Organic Synthesis*; Butterworths; London, 1981; pp 198–287. (b) Weber, W. P. *Silicon Reagents for Organic Synthesis*; Springer-Verlag: Berlin, 1983; pp 206–227. (c) Brownbridge, P. *Synthesis* **1983**, 1.

(2) Two procedures were recently published for such purpose: (a) Krafft, M. E.; Holton, R. A. *J. Am. Chem. Soc.* **1984**, *106*, 7619. (b) Kawanishi, M.; Itoh, Y.; Hieda, T.; Kozima, S.; Hitomi, T.; Kobayashi, K. *Chem. Lett.* **1985**, 647.

(3) Inoue, T.; Sato, T.; Kuwajima, I. *J. Org. Chem.* **1984**, *49*, 4671.

(4) Chlorotrimethylsilane has been found to accelerate the conjugate addition of Grignard reagents to afford the corresponding enol silyl ether directly. Horiguchi, Y.; Matsuzawa, S.; Nakamura, E.; Kuwajima, I., submitted for publication.

0022-3263/86/1951-1932$01.50/0