$$CrO^{2+} + CH_3OH + O_2 \rightarrow CrO_2^{2+} + CH_2O + 2H^+ \quad (2)$$

at 290 or 245 nm was fitted to an exponential function, yielding first-order rate constants that are linearly dependent on the concentration of methanol (inset to Figure 1) and independent of $[O_2]$ (0.26–1.3 mM). The slope, or bimolecular rate constant, is 22.7 ± 0.6 L mol^{-1} s^{-1} in 0.10 M $HClO_4$ at 25 °C. The intercept, 0.014 ± 0.001 s^{-1}, is real and reproducible and is attributed to the methanol-independent decomposition of the CrO^{2+}. Consistent with that, if methanol addition is delayed by a few minutes, the CrO_2^{2+} is no longer formed. The absorbance change for the reaction with methanol was used to estimate the yield of CrO^{2+}, ca. 50% based on total Cr.

The rate constant for the oxidation of methanol is independent of $[H^+]$ (0.02–1.0 M) at constant ionic strength but increases with ionic strength to 52.2 ± 1.4 L mol^{-1} s^{-1} at 1.0 M ionic strength. (The intercept also increases with ionic strength to 0.033 ± 0.001 s^{-1}.) A significant deuterium isotope effect was observed for CD_3OH (k_H/k_D = 3.2), implicating rate-determining carbon–hydrogen bond breaking. There was no effect of replacing the solvent H_2O by D_2O (which also converted CH_3OH to CH_3OD). Therefore the oxygen–hydrogen bond is not cleaved in the transition state, and the mechanism cannot be proton-assisted electron transfer.

The same reaction was observed for CrO^{2+} prepared from Cr^{2+} and $Tl^{III}(aq)$,[9] first mixed anaerobically in a 1:1 ratio and then added to oxygen-saturated aqueous methanol.[10] CrO^{2+} prepared in this way reacts with methanol with the same rate constant as the CrO^{2+} prepared from Cr^{2+} and O_2. As before, no CrO_2^{2+} forms without methanol; therefore we exclude the possibility that any Cr^{2+} remaining from the anaerobic step is the cause of the absorbance changes when O_2 is added. In the absence of a reductant such as methanol, no absorbance changes were seen in the UV spectrum; the CrO^{2+} simply decomposes to low-absorbing products. When methanol was present, formaldehyde was identified as a product of the reaction with CrO^{2+} by chromotropic acid analysis.[11] CrO^{2+} is also formed when Cr^{2+} and either CrO_2^{2+} or $CrOOCr^{4+}$ are mixed anaerobically. The anaerobic reactions of CrO^{2+} with methanol are not accompanied by absorbance changes due to formation of CrO_2^{2+}, and the Cr^{2+} product reacts rapidly with CrO^{2+}. Studies of other reactions are underway using the CrO^{2+} produced by these methods.

Further evidence that the oxidant is chromium(IV) is found in the methanol reaction, because CrO_2^{2+} is the inorganic product. Since the only known route to CrO_2^{2+} is the combination of oxygen with Cr^{2+}, we infer that it is the immediate product of the reaction. The only oxidant likely[12] to generate Cr^{2+} is Cr(IV):

$$CrO^{2+} + CH_3OH \xrightarrow{k} Cr^{2+} + CH_2O + 2H^+ \quad (3)$$

$$Cr^{2+} + O_2 \xrightarrow{\text{fast}} CrO_2^{2+} \quad (4)[6]$$

If the Cr(IV) is indeed an oxo species, then the two-electron oxidation of methanol is accomplished by hydride transfer,

$$[Cr{=}O\cdots H\cdots CH_2OH]^{2+}$$

followed by rapid proton equilibrations of $CrOH^+$ and CH_2OH^+. This mechanism accounts for the observed primary isotope effect and is thermodynamically more favorable than hydrogen atom transfer with formation of chromium(III) and an alkyl radical. Reduction of Cr(IV) to Cr^{2+} has an estimated potential of +0.84 V, with direct formation of the stable aldehyde, $E°(CH_3OH/CH_2O)$ = −0.23 V, whereas reduction of Cr(IV) to Cr^{3+} has an estimated potential of +2.0 V,[13] which is offset by the high potential of the radical, $E°(^{\bullet}CH_2OH/CH_3OH)$ = +1.29 V.[14] That is, the two-electron path is thermodynamically favored over the one-electron path by 0.26 V. Significant polarization of the carbon–hydrogen bond in the transition state leads to the creation of like charges, which is favored at higher ionic strengths. More studies of the chromium(IV) oxidant as a hydride acceptor and oxo-transfer agent are in progress.

Acknowledgment. This work was supported by a grant from the National Science Foundation. SLS received a 1967 Science and Engineering Fellowship from the Natural Sciences and Engineering Research Council of Canada.

(9) Dulz, G. E. Ph.D. Thesis, Columbia University, 1963.

(10) Oxygen is not present during the formation of the CrO^{2+} in the Tl(III) reaction with Cr^{2+}. In the subsequent reaction with methanol, the purpose of adding oxygen is to trap the Cr^{2+} product to yield CrO_2^{2+}, which serves as a spectroscopic probe.

(11) Bricker, C. E.; Johnson, H. R. *Anal. Chem.* **1945**, *17*, 40.

(12) Chromium(III) does not react with methanol; therefore the oxidant must have oxidation state >3. Chromium(VI) is known to react with alcohols, but very slowly.[5a] Chromium(V) may disproportionate more rapidly than it reacts with alcohols, but even a two-electron oxidation of methanol by chromium(V) will yield Cr(III), not Cr(II).

(13) Csanyi, L. J. In *Comprehensive Chemical Kinetics*; Bamford, C. H., Tipper, C. F. H., Eds.; Elsevier: Amsterdam, Vol. 7, p 510.

(14) Endicott, J. F. In *Concepts of Inorganic Photochemistry*; Adamson, A. W., Ed.; Wiley: New York, 1975; p 88.

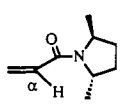
Control of Stereochemistry in Free Radical Reactions with Oxazolidine Auxiliaries

Ned A. Porter,* John D. Bruhnke, Wen-Xue Wu, Ian J. Rosenstein, and Robert A. Breyer

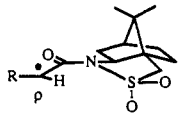
Department of Chemistry, Duke University Durham, North Carolina 27706

Received June 21, 1991

Recent reports[1-5] make it clear that control of acyclic stereochemistry is possible in free radical reactions. In the addition of radicals to chiral alkenes, for example, diastereofacial control is possible if the addition occurs α to a center substituted with an amide derived from C_2 symmetric pyrrolidines, e.g. **1**.[2] Stereochemical control in the addition of chiral radicals to unsaturated systems (ρ selectivity) is also possible if the radical is substituted with a C_2 symmetric pyrrolidine amide[3] or an imide derived from camphor sultam, e.g. **2**.[4] Other radical addition or atom transfer reactions exhibiting significant acyclic α or ρ stereoselectivity have also been recently reported.[5] Of the auxiliaries used to control acyclic stereochemistry, the dimethylpyrrolidine may give higher selectivities in some reactions but the sultam is commercially available and easy to remove after use.



1



2

For α or ρ selectivity with auxiliaries like the C_2 symmetric pyrrolidine or the sultam, the orientation about the C(α or ρ)–C(O) and the C(O)–N bond is critical in fixing the resident chiral group relative to the α or ρ center. For amides and imides, the *Z* orientation is favored for the C(α or ρ)–C(O) bond while C_2 symmetry avoids the need for C(O)–N rotamer control in di-

(1) Porter, N. A.; Giese, B.; Curran, D. P. *Acc. Chem. Res.* In press.

(2) (a) Porter, N. A.; Lacher, B.; Chang, V. H.; Magnin, D. R. *J. Am. Chem. Soc.* **1989**, *111*, 8309. (b) Porter, N. A.; Scott, D. M.; Lacher, B.; Giese, B.; Zeitz, H. G.; Lindner, H. J. *J. Am. Chem. Soc.* **1989**, *111*, 8311. (c) Porter, N. A.; Scott, D. M.; Rosenstein, I. J.; Giese, B.; Veit, A.; Zeitz, H. G. *J. Am. Chem. Soc.* **1991**, *113*, 1791. (d) Scott, D. M.; McPhail, A. T.; Porter, N. A. *Tetrahedron Lett.* **1990**, *31*, 1679. (e) Porter, N. A.; Wu, W.-X.; McPhail, A. T. *Tetrahedron Lett.* **1991**, *32*, 707.

(3) (a) Porter, N. A.; Swann, E.; Nally, J.; McPhail, A. T. *J. Am. Chem. Soc.* **1990**, *112*, 6740. (b) Giese, B.; Zehnder, M.; Roth, M.; Zeitz, H. G. *J. Am. Chem. Soc.* **1990**, *112*, 6741.

(4) Curran, D. P.; Shen, W.; Zhang, J.; Heffner, T. A. *J. Am. Chem. Soc.* **1990**, *112*, 6738.

(5) (a) Hamon, D. P. G.; Razzino, P.; Massy-Westropp, J.; Massy-Westropp, R. A. *J. Chem. Soc., Chem. Commun.* **1991**, *5*, 33. (b) Guindon, Y.; Lavallee, J.; Boisvert, L.; Chabot, C.; Delorme, D.; Yoakim, C.; Hall, D.; Lemieux, R.; Simoneau, B. *Tetrahedron Lett.* **1991**, *32*(1), 27. (c) Renaud, P.; Schubert, S. *Synlett* **1990**, 624. (d) Beckwith, A. L. J.; Chai, C. L. L. *J. Chem. Soc., Chem. Commun.* **1990**, *16*, 1087.

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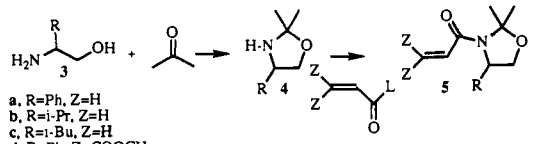
methylpyrrolidine, and dipole–dipole interactions presumably control the C(O)–N rotamer population for the sultam.

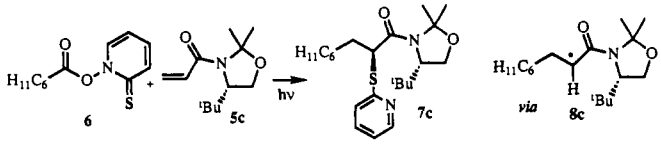
We report here that oxazolidines serve as excellent auxiliaries for control of α or ρ stereochemistry in free radical reactions.[6,7] These heterocycles have neither a C_2 nor a dipole–dipole rotamer control element and therefore represent a new auxiliary strategy for free radical acyclic stereoselection. Oxazolidines are readily prepared in one step from commercially available compounds and they are easily removed by acid hydrolysis. One of the oxazolidines gives the highest selectivities thus far reported in several free radical addition reactions.

The preparation of α,β unsaturated amides derived from oxazolidines or thiazolidines is straightforward.[7-9] The commercially available amino alcohols were stirred with acetone in dichloromethane/$MgSO_4$ overnight and the solutions filtered and concentrated. The oxazolidines were not purified but were directly acylated to give the target alkene in yields of 50–75% for the two-step sequence.



a, R=Ph, Z=H
b, R=i-Pr, Z=H
c, R=t-Bu, Z=H
d, R=Ph, Z=$COOCH_3$
e, R=i-Pr, Z=$COOCH_3$
f, R=t-Bu, Z=$COOCH_3$

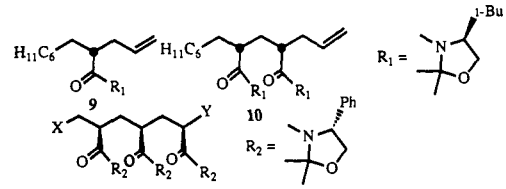
The first reaction examined to survey the potential of oxazolidine auxiliaries was the addition of alkyl radicals to the terminal end of acrylamides derived from oxazolidines and the trapping of the radicals so generated by *N*-hydroxypyridine-2-thione esters. This reaction was first utilized by Crich[10] in a study of acrylates and later by Giese[3b] to assess dimethylpyrrolidine as an auxiliary in ρ selectivity. Reaction of cyclohexyl *N*-hydroxypyridine-2-thione ester **6** with acrylamides **5a,b,c** produced the addition compounds **7a,b,c** in excellent yield and selectivity. At room temperature the



diastereoselectivities for formation of **7** from **5** were the following: **7a**, 33:1; **7b**, 7:1, **7c**, 60:1. For comparison, Giese observed selectivities of 14:1 at −35 °C for the dimethylpyrrolidine auxiliary in an analogous *N*-hydroxypyridine-2-thione ester trapping reaction.[3b] At −78 °C, selectivity for formation of **7a** was in excess of 70:1, indicating a typical temperature–selectivity dependence for radical addition.[3] The stereochemistry of product **7c** was established by single-crystal X-ray analysis.[11] Starting from **3c** having the *S* configuration, the configuration of the new stereogenic center formed in the reaction (ρ selection via **8c**) is *S*.

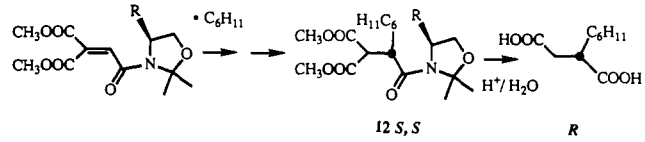
Addition of radicals like **8** to allyl stannanes or to acrylic acid derivatives was also examined to assess the efficacy of oxazolidines in ρ selectivity. Thus, reaction of the acrylamides **5a,b,c** with cyclohexyl iodide and allyltributylstannane at 80 °C[4] produced the addition products **9** and **10**. For R = *i*-Pr, selectivity in the addition reaction was modest (4:1), while for R = *t*-Bu, selectivity of the products formed having structure **9** was 25:1 and one major diastereomer having the structure **10** (tentative stereochemistry) is observed. For comparison, selectivities of 12:1 are observed for the Oppolzer sultam auxiliary (e.g. **2**) in an analogous allyl stannane transfer reaction.[4] The stereochemistry of **9** was established by independent synthesis.[12]

The acrylamides **5a** and **5b** were examined in telomerization reactions with bromotrichloromethane,[13] and these reactions proceed with significant diastereoselectivity (>15:1 for n = 3). For **5a**, the major n = 3 telomer is crystalline, and a single-crystal X-ray analysis[11] identified the stereochemistry of this diastereomer as shown in structure **11a**. The selectivity observed for the formation of **11a** indicates that not only alkene addition but also bromine atom transfer occurs stereoselectively.[13]



11 a, X=CCl_3, Y=Br
b, X=CH_3, Y=H

The standard reaction examined for α selectivity in the addition of radicals to chiral alkenes was the addition of cyclohexyl radical to the alkenes **5d–f**. Addition of radicals to these trisubstituted alkenes occurs at the monosubstituted end of the olefin to give the addition products **12**. The diastereoselectivity observed for addition of cyclohexyl radical to these alkenes at 0 °C is the following: **5d**, 1.1:1; **5e**, 10:1; **5f**, > 80:1. Addition of cyclohexyl radical under similar conditions to a trisubstituted alkene with the C_2 dimethylpyrrolidine auxiliary gives a selectivity of 40:1.[2b] Hydrolysis of the addition products **12** in dioxane/1 N HCl shows



that starting from **5e** or **5f** having the *S* configuration, the configuration of the new stereogenic center in the major diastereomer of **12** formed in the reaction (α stereoselection) is *S*.

Two C(O)–N rotamers can be observed by NMR for alkenes **5a–f**, and NOE experiments suggest that the *Z* rotamer is favored for R = Ph (*Z*:*E* > 10:1) or *i*-Pr (*Z*:*E* 6.5–9:1). For R = *t*-Bu, the auxiliary that gives the highest selectivity in all of the reactions examined, the rotamer ratio is only 2:1. Clearly rotamer population control is not the deciding factor in stereoselectivity.[14] The net α selectivity observed for alkenes substituted with oxazolidines depends on the reactivity and selectivity of oxazolidine rotamers while the net ρ selectivity observed is the result of a kinetically complex system that includes the rate of formation, rate of interconversion, rate of addition, and selectivity of radical oxazolidine rotamers.[14]

Molecular mechanics calculations suggest that for R = *t*-Bu this group assumes a pseudoaxial orientation in the half-chair oxazolidine for both rotamers because of steric crowding with the α or ρ center (*Z* rotamer) or the amide carbonyl (*E* rotamer). There is significant pyramidalization of the amide N. This dis-

(6) For a discussion of the use of other heterocyclic auxiliaries in carbanion chemistry, see: (a) Evans, D. A. *Asymmetric Synthesis*; Morrison, J. D., Ed.; Academic Press: Orlando, 1984; p 2. (b) Lumtowski, K. A.; Meyers, A. I. *Asymmetric Synthesis*; Morrison, J. D., Ed.; Academic Press: Orlando, 1984; Vol. 3, p 218. (c) Seebach, D. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 1624. (d) Mukaiyama, T. *Challenges in Synthetic Organic Chemistry*; Int. Ser. Monographs on Chemistry, No. 20. Oxford University Press: Oxford, 1990. (e) Rawson, D. J.; Meyers, A. I. *J. Org. Chem.* **1991**, *56*, 2292.

(7) Stereoselective reactions of an oxazolidine substituted metal carbene have been reported. Imwinkelried, R.; Hegedus, L. S. *Organometallics* **1988**, *7*, 702.

(8) For a related auxiliary, see: Solodin, I.; Goldberg, Y.; Zelcans, G.; Lukevics, E. *J. Chem. Soc., Chem. Commun.* **1990**, 1321.

(9) We have examined analogous thiazolidines derived from cysteine and find significant diastereoselectivity in the Barton ester trapping reaction.

(10) Crich, D.; Davies, J. W. *Tetrahedron Lett.* **1987**, *28*, 2205.

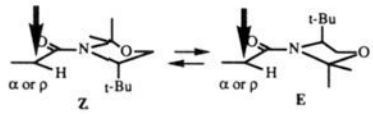
(11) X-ray analyses were carried out by A. T. McPhail at Duke University and the details of these analyses will be published in due course.

(12) The amide **9** was prepared via the corresponding carboxylic acid. This carboxylic acid was prepared enriched in the *S* enantiomer by allylation of the enolate prepared from a proline amide precursor. The stereochemistry of **9** was also correlated with that of an analogous compound prepared (ref 4) having an Oppolzer sultam auxiliary in place of the oxazolidine.

(13) Stereoselective halogen atom transfer has also been observed with the dimethylpyrrolidine auxiliary: Porter, N. A.; Breyer, R.; Swann, E.; Nally, J.; Allen, T.; McPhail, A. T. *J. Am. Chem. Soc.* In press.

(14) Seeman, J. I. *Chem. Rev.* **1983**, *83*, 83. For a recent discussion of distortion and reactivity of 5-membered-ring heterocycles with *t*-Bu substituents, see: Seebach, D.; Maetzke, T.; Petter, W.; Klotzer, B.; Plattner, D. A. *J. Am. Chem. Soc.* **1991**, *113*, 1781.

tortion results in a pseudoaxial methyl group at C-2 protecting the same α or ρ face in the *E* rotamer that the *t*-Bu group protects in the *Z* rotamer. We suggest that for R = *t*-Bu both alkene or radical C(O)-N rotamers must favor the formation of the same product diastereomer, while this bias is apparently not guaranteed for other R substituents (e.g. α selectivity for R = Ph is 1.1:1).



The ease of preparation of the oxazolidines and the high selectivities observed for these auxiliaries make them attractive candidates for further study. It is noteworthy that the *t*-Bu-substituted compound gives the highest selectivity reported to date in a variety of free radical addition reactions.

Acknowledgment. Support for this research from NIH (HL17921) and NSF is gratefully acknowledged. J.D.B. and I.J.R. thank the Burroughs Wellcome Foundation for support. We thank Professor D. P. Curran for a sample used to correlate the stereochemistry of **9** with products formed in Curran's labs, ref 4.

Supplementary Material Available: Experimental details for acrylamide and trisubstituted alkene preparations and free radical addition reactions (3 pages). Ordering information is given on any current masthead page.

Intramolecular Reaction of 1,5-Disila-1,4-pentadiene: Formation of 2,4,5-Trisilabicyclo[1.1.1]pentane and 2,3,5-Trisilabicyclo[2.1.0]pentane

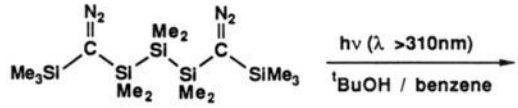
Wataru Ando,* Hitoaki Yoshida, Katsuaki Kurishima, and Mitsuru Sugiyama

Department of Chemistry, University of Tsukuba Tsukuba, Ibaraki 305, Japan

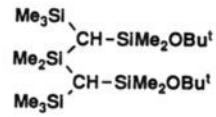
Received March 11, 1991

The recent surge of interest in strained compounds has resulted in reports of constructing polycyclic system with group 14 elements, Si, Ge, and Sn.[1] A bulky substituent is indispensable to the usual method, since the methodology is based on the intermolecular oligomerization reaction. Our recent studies of silene chemistry via silyldiazomethane[2] encouraged the synthesis of the polysilabicyclo system by new methodology. We report here the fruitful procedure for the formation of strained compounds by intramolecular reaction of 1,5-disila-1,4-diene **2**.

Benzene solution of bis(silyldiazomethyl) compound **1a**[3] with excess ^{t}BuOH was allowed to irradiate for 4.5 h, producing **3** in 47% yield, which is the 2 mol of ^{t}BuOH adduct (eq 1).[6] This result shows that **1**,[3] upon irradiation, serves as a disiladiene synthon and raises, the possibilities of intramolecular reaction in the absence of a trapping reagent.



1a



(1)

3

The photolysis of **1** without trapping reagent yielded mainly polymeric compounds, with the main interest compound, trisilabicyclo[1.1.1]pentane **4**: yield **4a**, 0.39%,[7] and **4b**, 1.4%.[8,9]

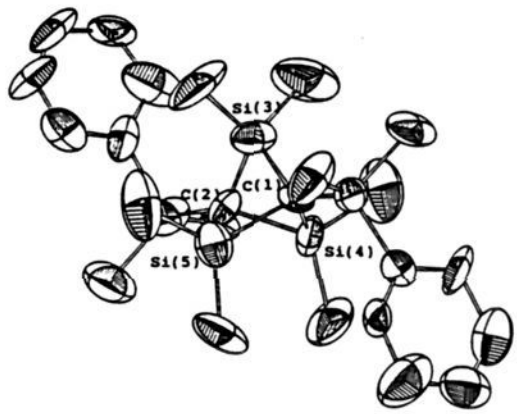


Figure 1. X-ray structure of **4b**. Selected bond lengths (Å) and angles (deg) are as follows: Si(3)-C(1), 1.92; Si(4)-C(1), 1.95; Si(5)-C(1), 1.97; Si(3)-C(2), 1.84; Si(4)-C(2), 1.90; Si(5)-C(2), 1.90; Si(3)-Si(4), 2.54; C(1)-Si(3)-C(2), 81.1; C(1)-Si(4)-C(2), 78.9; C(1)-Si(5)-C(2), 78.2; Si(3)-C(1)-Si(4), 82.1.

Chart I

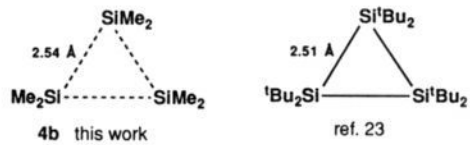
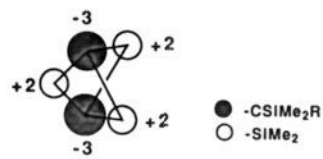


Chart II



(1) Scheldrick, W. S. In *The Chemistry of Organic Silicon Compounds*, Patai, S., Rappoport, Z.; Ed.: John Wiley & Sons Ltd., 1989; Part 1, Chapter 3. Bock, H. *Angew. Chem., Int. Ed. Engl.* **1989**, *28*, 1627. Sita, L. R.; Kinoshita, I. *J. Am. Chem. Soc.* **1990**, *112*, 8839. Sita, L. R.; Kinoshita, I. *Organometallics* **1990**, *9* 2865 and references cited therein. Sekiguchi, A.; Kabuto, C.; Sakurai, H. *Angew. Chem., Int. Ed. Engl.* **1989**, *28*, 55.

(2) (a) Ando, W.; Hagiwara, T.; Migita, M. *J. Am. Chem. Soc.* **1973**, *95*, 7518. (b) Ando, W.; Sekiguchi, A.; Migita, T.; Kammula, S.; Green, M.; Jones, M., Jr. *J. Am. Chem. Soc.* **1975**, *97*, 3818. (c) Sekiguchi, A.; Ando, W. *J. Am. Chem. Soc.* **1981**, *103*, 3579. (d) Ando, W.; Sekiguchi, A.; Sato, T. *J. Am. Chem. Soc.* **1981**, *103*, 5573. (e) Sekiguchi, A.; Ando, W. *J. Am. Chem. Soc.* **1984**, *106*, 1486.

(3) The starting material **1** was prepared in 83% (R = Me) and 85% (R = Ph) crude yield, respectively.[4] An analytically pure sample was obtained by alumina column chromatography and GPC. **1a**: yellow oil; MS *m/e* 400 (M^+); ^{1}H NMR (C_6D_6) δ 0.340 (s, 6 H), 0.297 (s, 12 H), 0.126 (s, 18 H); IR (neat) ν/cm^{-1} 2042 (C=N$_2$). **1b**: yellow oil; MS *m/e* 524 (M^+); ^{1}H NMR (C_6D_6) δ 7.27-7.17 (m, 10 H), 0.36 (s, 12 H), 0.26 (s, 16 H), 0.17 (s, 12 H); IR (neat) ν/cm^{-1} 2042 (C=N$_2$).

(4) (a) Sekiguchi, A.; Ando, W. *Organometallics* **1987**, *6*, 1857. (b) Ando, W.; Sekiguchi, A.; Migita, T. *Chem. Lett.* **1976**, 779. (c) Sekiguchi, A.; Ando, W. *Chem. Lett.* **1986**, 2025.

(5) Ando, W. IXth International Symposium on Organosilicon Chemistry, Edinburg, I-17, 1990.

(6) The product **3** was isolated by preparative GLC. **3**: MS *m/e* 477 (M-15); ^{1}H NMR (C_6D_6) δ 1.48 (s, 18 H), 0.45 (br s, 18 H), 0.34 (s, 18 H), 0.24 (s, 2 H).

(7) THF was used as solvent. **4a**: colorless crystals; MS *m/e* 344 (M^+); ^{1}H NMR (C_6D_6) δ 0.44 (s, 18 H), 0.06 (s, 18 H); ^{13}C NMR (C_6D_6) δ 17.03 (s), 7.56 (q), 1.98 (q); ^{29}Si NMR (C_6D_6) δ -4.9 ($SiMe_2$), -11.4 ($SiMe_3$); exact MS found 329.1403, calcd for $C_{13}H_{33}Si_5$ (M - 15) 329.1428.

(8) Cyclohexane was used as solvent for routine work and *n*-hexane-d_{14} for low-temperature NMR study. For the reaction of **1b**, UV-35 filter was used to cut the light of λ < 350 nm.

(9) **4b**: colorless crystals; mp 128-129 °C; MS *m/e* 453 (M - 15); ^{1}H NMR (C_6D_6) δ 7.5-7.1 (m, 10 H), 0.30 (s, 12 H), 0.29 (s, 18 H); ^{13}C NMR (C_6D_6) δ 142.0 (s), 133.4 (d), 128.0 (d), 126.7 (d), 7.36 (q), 0.11 (q); ^{29}Si NMR (C_6D_6) δ 31.9, -51.0; exact MS found 468.1952, calcd for $C_{24}H_{40}Si_5$ 468.1976.

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