246 (M^+), 244 (M^+), 228, 226, 209; HR-MS (M^+) calcd for $C_{13}H_{21}O_2Cl$ 244.1231, found 244.1228.

(4*S*)-2-Chloro-4-hydroxy-4-[2(*Z*)-octenyl]-2-cyclopenten-1-one (19). To a stirred solution of **16** (332 mg, 1.38 mmol) in CH_2Cl_2 (19 mL) were successively added pyridine (3.6 mL), methanesulfonic anhydride (307 mg, 1.76 mmol), and 4-(dimethylamino)pyridine (33 mg, 0.27 mmol) in an ice bath. After being stirred for an additional 40 min, the reaction was quenched with ether (20 mL) and filtered. To the filtrate was added toluene (50 mL), and the solvents were evaporated in vacuo to give **17** as a pale yellow oil, to which was added 85% aqueous DMSO (20 mL). The resulting solution was stirred at 23 °C for 24 h and then diluted with ethyl acetate. The organic layer was successively washed with saturated aqueous $NaHCO_3$ and brine, dried ($MgSO_4$), and concentrated to give an oily residue **18**, to which was added DMF (3 mL) and pyridinium dichromate (800 mg, 2.13 mmol) in an ice bath. Stirring was continued at the same temperature for 15 h, and the reaction mixture was poured into water. Extraction with ether and evaporation of the dried solvent ($MgSO_4$) in vacuo gave the oily residue, which was purified by silica gel column chromatography (toluene–ethyl acetate, 10:1) to give **19** (169 mg, 51%) as a nearly colorless oil: ^1H NMR ($CDCl_3$) δ 0.90 (t, J = 5 Hz, 3 H), 1.10–1.57 (m, 6 H), 1.87–2.33 (m, 3 H), 2.57 (d, J = 6 Hz, 2 H), 2.67 (d, J =3 Hz, 2 H), 5.23–5.97 (m, 2 H), 7.38 (s, 1 H); IR (neat) 3350, 1730, 1600 cm^{-1}; mass spectrum, m/e 244 (M^+), 242 (M^+), 226, 224; HR-MS (M^+) calcd for $C_{13}H_{19}O_2Cl$ 242.1073, found 242.1084.

(4*S*)-2-Chloro-4-(methoxymethoxy)-4-[2(*Z*)-octenyl]-2-cyclopenten-1-one (20). A solution of **19** (18 mg, 0.074 mmol), diisopropylethylamine (0.13 mL, 0.74 mmol), and chloromethyl methyl ether (28 μL 0.37 mmol) was stirred at 60 °C for 4 h in a sealed tube. The reaction mixture was diluted with ether, and the ether layer was washed with H_2O. Concentration of the dried solvent ($MgSO_4$) afforded the oily residue, which was purified by silica gel column chromatography (hexane–ether, 8:1) to give **20** (18.3 mg, 86%) as a nearly colorless oil: $[\alpha]^{20}{}_D$ +50.2° (c 0.315, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 0.89 (t, J = 6.74 Hz, 3 H), 1.20–1.40 (m, 6 H), 2.01 (m, 2 H), 2.48–2.63 (m, 2 H), 2.59 (d, J = 18.8 Hz, 1 H), 2.79 (d, J = 18.8 Hz, 1 H), 3.37 (s, 3 H), 4.63 (d, J = 7.6 Hz, 1 H), 4.71 (d, J = 7.6 Hz, 1 H), 5.34 (m, 1 H), 5.59 (m, 1 H), 7.40 (s, 1 H); IR (neat) 1735, 1600 cm^{-1}; mass spectrum, m/e 288 (M^+), 286 (M^+), 227, 225, 177, 176, 175, 151, 149, 141, 124; HR-MS (M^+) calcd for $C_{15}H_{23}O_3Cl$ 286.1336, found 286.1336.

Efficient Synthesis of Silyl Azides Using Sodium Azide Impregnated on Amberlite XAD Resin

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Silyl azides,[1] which are useful reagents in organic synthesis, have generally been prepared by the reaction of silyl chlorides with LiN_3[2] or NaN_3.[3] However, most of these methods require a long reaction time, and LiN_3 is not commonly available. Among these methods, although the reaction of silyl chloride with NaN_3 in hexamethylphosphoric triamide (HMPT) or *N*,*N*-dimethylformamide (DMF) is more general,[3c] HMPT has been reported to be a potent chemical carcinogen, and DMF can react with the product, silyl azide. Furthermore, this method cannot be applied to the preparation of silyl azides possessing an Si—H or Si—C=CH_2 bond from the corresponding silyl chlorides.[3c]

Table I. Reaction of Me_3SiCl with NaN_3/XAD-4 in Various Solvents[a]

solvent	temp, °C	time, h	yield,[b] %
CH_3CN	40	<1	>96[c]
DME[d]	40	4	>96[c]
CH_2Cl_2	40	6	>98[c]
benzene	60	2	85
	60	6	>98[c]
hexane	60	2	85
	60	6	>98[c]
decalin	60	6	>98[c]
none	40	6	67

[a] All reactions were carried out with Me_3SiCl (7.5 mmol) and NaN_3 (15 mmol)/XAD-4 (3.75 g). [b] Determined by GLC with toluene as an internal standard. [c] Almost complete conversion was observed. [d] 1,2-Dimethoxyethane.

In previous papers, it has been shown that the impregnation of anionic species on Amberlite XAD resin (XAD resin) enhances the substitution reaction toward alkyl halides,[4] and the reaction of silyl chloride with KCN or NaCN impregnated on XAD resin (KCN or NaCN/XAD) gives the corresponding silyl cyanides in good yields under mild conditions.[5] This paper describes that a large number of silyl azides, including silyl azides having a Si—H or Si—CH=CH_2 bond, can be prepared by the reaction of the corresponding silyl chlorides with NaN_3 impregnated on XAD resin (NaN_3/XAD) in good yields under mild conditions with common solvents.

$$R_{4-n}SiCl_n \xrightarrow{NaN_3/XAD} R_{4-n}Si(N_3)_n$$

Results and Discussion

In order to elucidate the solvent effect, the reaction was performed with trimethylsilyl chloride (Me_3SiCl) and NaN_3/XAD-4 in various solvents. Table I shows the results. In acetonitrile, the reaction was very fast and gave trimethylsilyl azide (Me_3SiN_3) in greater than 96% yield (~100% conversion) within 1 h. Interestingly, even in low-polar solvents, which were not suitable for the reaction of Me_3SiCl with KCN or NaCN/XAD,[5] the complete conversion of Me_3SiCl was observed after relatively short reaction times, and even the reaction without any solvent gave 67% yield of Me_3SiN_3 at 40 °C after 6 h. These results suggest that various solvents can be utilized for the reaction.

On the basis of the results, other silyl azides were prepared (Table II). In general, silyl azides were obtained in high yields under mild conditions. It is worth noting that silyl azides having a Si—H or Si—CH=CH_2 bond were obtained in high yields directly from the corresponding silyl chlorides. Previously reported methods could not produce these compounds directly from the corresponding silyl chlorides.[3c] R_2HSiN_3 was previously prepared by an exchange reaction between Me_3SiN_3 and R_2HSiCl.[6]

$$R_2HSiCl + Me_3SiN_3 \rightarrow R_2HSiN_3 + Me_3SiCl$$

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Table II. Preparation of Alkylsilyl Azides[a]

silyl chloride	molar ratio[b]	solvent	temp, °C	time, h	product	yield,[c] %	bp, °C (mmHg) found	bp, °C (mmHg) lit.	IR (CCl_4), cm^{-1}
Me_3SiCl	2.0	decalin	60	6	Me_3SiN_3	82 (>98)	92-97 (760)	95-97 (760)[3c]	2140
Et_3SiCl	2.0	CH_2Cl_2	40	6	Et_3SiN_3	87 (>95)	97-99 (80)	104 (95)[2c]	2135
	1.5	CH_2Cl_2	40	8		(~84)			
$Ph_2MeSiCl$	2.0	CH_2Cl_2	40	5	Ph_2MeSiN_3	89 (>98)	96-98 (0.2)	114 (1)[6]	2143
	1.5	CH_3CN	40	4		(>98)			
$PhMe_2SiCl$	1.5	CH_2Cl_2	40	5	$PhMe_2SiN_3$	(~90)	89-90 (11)	90 (11)[6]	2140
	2.0	CH_2Cl_2	40	5		88 (>98)			
t-$BuMe_2SiCl$	2.0	CH_2Cl_2	40	20	*t*-$BuMe_2SiN_3$	89 (~98)	88-91 (110)	78 (67)[8]	2140
	2.5	CH_2Cl_2	40	6		85 (~98)			
Me_2SiCl_2	4.0	CH_2Cl_2	40	6	$Me_2Si(N_3)_2$	80 (>98)	73-76 (60)	144-145 (760)[3c]	2142, 2163
Ph_2SiCl_2	2.0	CH_3CN	40	4	$Ph_2Si(N_3)_2$	76 (~98)	110-114 (0.08)	121-123 (0.1)[3c]	2146, 2164
Ph_2HSiCl	2.0	CH_2Cl_2	40	5	Ph_2HSiN_3	84 (>98)	106-109 (0.4)	139 (2)[6]	2145
$PhMeHSiCl$	2.0	CH_2Cl_2	40	6	$PhMeHSiN_3$	80 (~98)	85-88 (13)	95 (15)[6]	2140
$Ph_2(CH_2{=}CH)SiCl$	2.0	CH_2Cl_2	40	7	$Ph_2(CH_2{=}CH)SiN_3$	28, 90[d]	120-125 (0.7)		2140

[a] All reactions were carried out with NaN_3 (30 mmol)/XAD-4 (8.6 g) and a solvent (40 mL). [b] Molar ratio NaN_3/silyl chloride. [c] Isolated yield. Values in parentheses are the yields determined by GLC. [d] Isolated as the triphenylphosphinimine derivative.

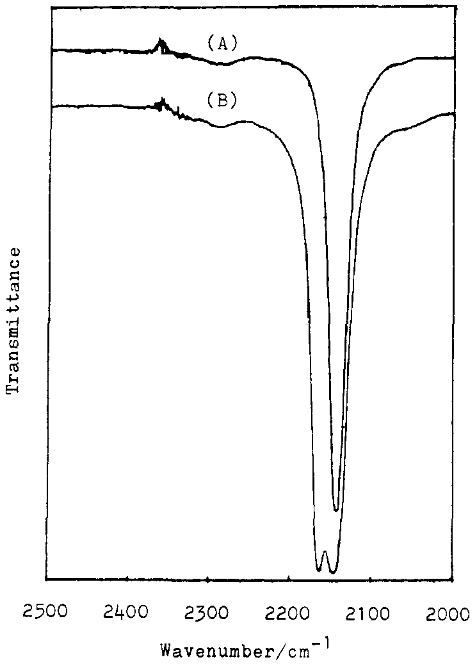

Figure 1. Plot of time vs molar fractions of Ph_2SiHN_3, Ph_2SiH_2, and $Ph_2Si(N_3)_2$. The reaction was carried out with NaN_3 (15 mmol)/XAD-4 (4.3 g) and Ph_2SiHCl (10 mmol) in CH_3CN (20 mL) at 40 °C. (○) Ph_2SiHN_3, (□) Ph_2SiH_2, (△) $Ph_2Si(I_3)_2$.

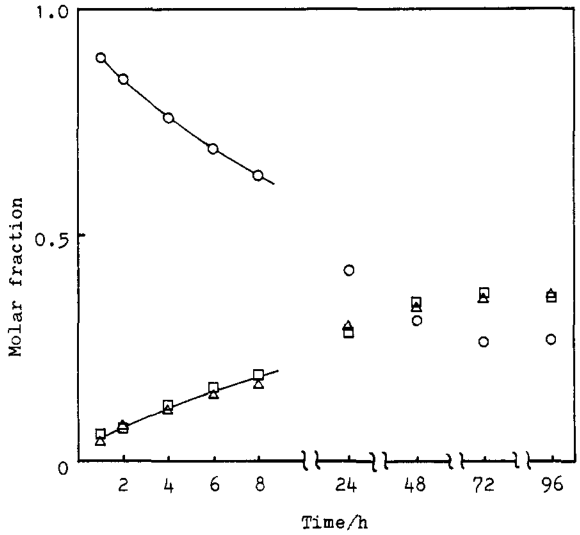

Figure 2. IR spectra of (A) Ph_2MeSiN_3 and (B) $Ph_2Si(N_3)_2$.

The infrared absorption band of the asymmetric azide stretching for silyl monoazide appears at ca. 2140 cm^{-1}. In the case of silyl diazide, however, the absorption band split into two bands (Table II and Figure 1). Since no split absorption band was observed for alkyl *gem*-diazides,[1b] it seems that this split is characteristic of silyl diazide and the two azides groups are not equal to each other. This is probably due to the interaction between d orbitals of silicon and azide groups.

In the reaction of Ph_2HSiCl with NaN_3/XAD-4, when CH_2Cl_2 was used as a solvent, Ph_2HSiN_3 was obtained in greater than 98% yield (~100% conversion) at 40 °C after 5 h. When acetonitrile was employed as the solvent, however, Ph_2SiH_2 and $Ph_2Si(N_3)_2$ were produced in substantial yields. Figure 2 shows the relationship between reaction time and molar fractions of Ph_2HSiN_3, Ph_2SiH_2, and $Ph_2Si(N_3)_2$. The amount of Ph_2HSiN_3 decreased and the amount of Ph_2SiH_2 and $Ph_2Si(N_3)_2$ increased with increasing reaction time. This conversion attained equilibrium after ca. 72 h, and the molar fractions of Ph_2SiH_2 and $Ph_2Si(N_3)_2$ were substantially identical. When DMF was used as the solvent for this reaction, the equilibrium was reached within 2 h. This conversion rate was in the order of CH_2Cl_2 ≪ acetonitrile ≪ DMF. On the other hand, although Ph_2HSiN_3 was stirred at 40 °C for 24 h with or without ZnI_2 in acetonitrile, no change in the amount of Ph_2HSiN_3 and no production of Ph_2SiH_2 and $Ph_2Si(N_3)_2$ were observed. These findings indicate that the disproportionation of Ph_2HSiN_3 to Ph_2SiH_2 and $Ph_2Si(N_3)_2$ is catalyzed by N_3^- in the bulk organic phase.

$$2Ph_2HSiN_3 \xrightarrow{N_3^-} Ph_2SiH_2 + Ph_2Si(N_3)_2$$

Presumably this disproportionation prevents the previously reported methods using dipolar solvents[3c] from producing silyl azides having a Si-H bond directly from the corresponding silyl chlorides.

Experimental Section

Materials. All the solvents were dried with molecular sieves 4A. Alkylsilyl chlorides were used as obtained. Diphenylsilane (Ph_2SiH_2) was prepared by the reaction of Ph_2HSiCl with $LiAlH_4$ in diethyl ether (84% yield, bp 73-75 °C/0.5 mmHg, lit.[7] bp 75-76 °C/0.5 mmHg). NaN_3/XAD-4 was prepared by the method similar for KCN or NaCN/XAD:[5] Amberlite XAD-4 (4.3 g) was

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added to a solution of NaN_3 (15 mmol) in 40% aqueous methanol (5 g), and the resulting mixture was allowed to stand for ca. 20 min. The solvent was removed by vigorous shaking under reduced pressure ($\sim$20 mmHg) at ambient temperature to 90 °C. The half-dried NaN_3/XAD-4 reagent was further dried at 90 °C for 4 h under vacuum ($\sim$0.05 mmHg).

In all reactions, alkylsilyl chloride and a solvent were directly added to the flask in which the NaN_3/XAD-4 was made and dried.

Reaction of Me_3SiCl with NaN_3/XAD-4 in Various Solvents. A mixture of NaN_3 (15 mmol)/XAD-4 (3.75 g), Me_3SiCl (7.5 mmol), and a solvent (20 mL) was stirred for the indicated time and at the indicated temperature. After the mixture had been cooled to room temperature, it was analyzed by GLC with toluene as an internal standard (Silicon OV-17, 10%, 3 m × 3 mm, 40 or 50 °C).

Me_3SiN_3: A mixture of Me_3SiCl (1.62 g, 15 mmol), NaN_3 (2.00 g, 30 mmol)/XAD-4 (8.6 g), and decalin (40 ml) was stirred at 60 °C for 6 h. GLC showed that the conversion of Me_3SiCl was $\sim$100%. The reaction mixture was distilled directly by using a Vigreux column (10 cm), giving 1.41 g (82% yield, 98% purity) of Me_3SiN_3, bp 92–97 °C (760 mmHg) [lit.[3c] bp 95–97 °C (760 mmHg)]. IR (CCl_4): 2140 cm^{-1}.

Diphenylmethylsilyl Azide (Ph_2MeSiN_3): A mixture of diphenylmethylsilyl chloride ($Ph_2MeSiCl$) (3.50 g, 15 mmol), NaN_3 (2.00 g, 30 mmol)/XAD-4 (8.6 g), and CH_2Cl_2 (40 mL) was stirred for 5 h at 40 °C. GLC showed the complete conversion of $Ph_2MeSiCl$ (Silicone SE-30, 10%, 1 m × 3 mm, 190 °C). The solid material was filtered and washed with CH_2Cl_2 (60 mL). After removal of the solvent, the residue was distilled under vacuum, giving 3.18 g (89%) of Ph_2MeSiN_3, bp 96–98 °C (0.2 mmHg) [lit.[6] bp 114 °C (1 mmHg)]. IR (CCl_4): 2143 cm^{-1}.

Dimethylsilyl Diazide ($Me_2Si(N_3)_2$): A mixture of dimethylsilyl dichloride (Me_2SiCl_2) (0.97 g, 7.5 mmol), NaN_3 (2.00 g, 30 mmol)/XAD-4 (8.6 g), and CH_2Cl_2 (40 ml) was stirred for 6 h at 40 °C. The same treatment used with Ph_2MeSiN_3 was performed. After removal of the solvent at room temperature and 160–200 mmHg, the residue was distilled with use of a Vigreux column (15 cm) under reduced pressure, giving 0.86 g (80% yield, 98% purity) of $Me_2Si(N_3)_2$, bp 73–76 °C (60 mmHg) [lit.[3c] bp 144–145 °C (760 mmHg)]. IR (CCl_4): 2163 and 2142 cm^{-1}.

***tert*-Butyldimethylsilyl Azide (t-$BuMe_2SiN_3$):** A mixture of *tert*-butyldimethylsilyl chloride (t-$BuMe_2SiCl$) (1.96 g, 12 mmol), NaN_3 (2.00 g, 30 mmol)/XAD-4 (8.6 g), and CH_2Cl_2 (40 mL) was stirred at 40 °C for 6 h. GLC showed the complete conversion of the silyl chloride. The same treatment used with Ph_2MeSiN_3 was performed. After removal of the solvent at room temperature and 160–200 mmHg, the residue was distilled under reduced pressure, giving 1.60 g (85%) of t-$BuMe_2SiN_3$, bp 90–91 °C (110 mmHg) [lit.[8] bp 78 °C (67 mmHg)]. IR (CCl_4): 2140 cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.220 (s, 6 H), 0.950 (s, 9 H). MS (m/z): 157 (M^+), 142, 128, 100.

***P,P,P*-Triphenyl-*N*-(*tert*-butyldimethylsilyl)phosphinimine (t-$BuMe_2SiN{=}PPh_3$).** A mixture of t-$BuMe_2SiN_3$ (0.63 g, 4 mmol), triphenylphosphine (PPh_3) (0.94 g, 3.6 mmol), and toluene (4 ml) was refluxed for 16 h. GLC showed the complete conversion of PPh_3. The insoluble material was filtered off, and the solvent was removed under reduced pressure to give residual solid (1.38 g, 98% yield), recrystallization of which from toluene gave 1.20 g (85%) of t-$BuMe_2SiN{=}PPh_3$, mp 81.0–82.5 °C. IR (CCl_4): 1434, 1244 (N=P), 1108 cm^{-1}. ^1H NMR ($CDCl_3$): δ −0.171 (s, 6 H), 0.811 (s, 9 H), 7.427–7.809 (m, 15 H). Anal. Calcd for $C_{24}H_{30}NPSi$: C, 73.62; H, 7.72; N, 3.58. Found: C, 73.75; H, 7.55; N, 3.56. MS (m/z): 376 ($M^+ - CH_3$), 334, 277.

Diphenylvinylsilyl Azide ($Ph_2(CH_2{=}CH)SiN_3$). A mixture of diphenylvinylsilyl chloride ($Ph_2(CH_2{=}CH)SiCl$) (3.67 g, 15 mmol), NaN_3 (2.00 g, 30 mmol)/XAD-4 (8.6 g), and CH_2Cl_2 (40 mL) was stirred for 7 h at 40 °C. GLC showed the complete conversion of the silyl chloride and the sole product (Silicone SE-30, 10%, 1 m × 3 mm, 190 → 280 °C). Removal of the solvent gave 3.69 g of the residue, which was distilled under vacuum to give only 0.98 g (28% yield) of $Ph_2(CH_2{=}CH)SiN_3$, because of the decomposition of the silyl azide during distillation. Bp 120–125 °C (0.7 mmHg). IR (CCl_4): 2140 cm^{-1}. ^1H NMR ($CDCl_3$): δ 5.717–6.579 (m, 3 H), 7.353–7.740 (m, 10 H). MS (m/z): 251 (M^+), 224, 209, 183.

***P,P,P*-Triphenyl-*N*-(diphenylvinylsilyl)phosphinimine ($Ph_2(CH_2{=}CH)SiN{=}PPh_3$).** A mixture of $Ph_2(CH_2{=}CH)SiCl$ (1.84 g, 7.5 mmol), NaN_3 (1.00 g, 15 mmol)/XAD-4 (4.3 g), and CH_2Cl_2 (20 mL) was stirred at 40 °C for 7 h. GLC showed the complete conversion of the silyl chloride. After the same treatment used with Ph_2MeSiN_3 was performed, removal of the solvent gave 1.86 g ($\sim$100% yield) of the crude $Ph_2(CH_2{=}CH)SiN_3$, which was heated with PPh_3 (1.95 g, 7.5 mmol) under reflux in benzene (20 mL) for 16 h. The insoluble material was filtered off, and the solvent was removed to give the solid residue, which was washed with benzene–hexane and dried under reduced pressure, giving 3.14 g (90% overall yield). This product was recrystallized from benzene to give pure $Ph_2(CH_2{=}CH)SiN{=}PPh_3$, mp 144–145 °C. IR ($CCl_4$): 1436, 1256 (N=P), 1110 cm^{-1}. ^1H NMR ($CDCl_3$): δ 5.815–5.991 (m, 3 H), 7.22–7.848 (m, 25 H). MS (m/z): 485 (M^+), 458, 408, 381, 334.

Diphenylsilyl Azide (Ph_2HSiN_3). A mixture of diphenylsilyl chloride (Ph_2HSiCl) (1.64 g, 7.5 mmol), NaN_3 (1.00 g, 15 mmol)/XAD-4 (4.3 g), and CH_2Cl_2 (20 ml) was stirred at 40 °C for 5 h. GLC showed the complete conversion of the silyl chloride (Silicone SE-30, 10%, 1 m × 3 mm, 180 °C). After the same treatment used with Ph_2MeSiN_3, removal of the solvent gave 1.66 g of the crude product, distillation of which gave 1.43 g (84% yield) of Ph_2HSiN_3, bp 106–109 °C (0.4 mmHg) [lit.[6] bp 139 °C (2 mmHg)]. IR (CCl_4): 2145 cm^{-1}.

Reaction of Ph_2HSiCl with NaN_3/XAD-4 in Acetonitrile. A mixture of Ph_2HSiCl (2.19 g, 10 mmol), NaN_3 (1.00 g, 15 mmol)/XAD-4 (4.3 g), acetonitrile (20 mL), and pentadecane as an internal standard was stirred at 40 °C. The reaction mixture was analyzed by GLC at various intervals (Silicone SE-30, 10%, 1 m × 3 mm, 180 °C).

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Phenyl Sulfone-Directed Diastereoselective Cyclization of an Epoxy Allylsilane System

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In order to prepare a trimethylsilyl-substituted cyclization substrate for a planned biomimetic-type total synthesis of the secotrinervitane diterpenes,[1] we required a new 2-[(trimethylsilyl)methyl]allyl anion synthon[2] which would allow *two* sequential alkylations to be performed. An intramolecular epoxy allylsilane cyclization was envisioned to complete the carbon skeleton. Epoxy allylsilane cyclizations were introduced by Fleming,[3] and their utility has been extended by the Weiler,[4a] Procter,[4b] and Chan[4c]

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