7a, 117679-38-6; 7b, 117679-48-8; 8a, 117679-39-7; 8b, 117679-49-9; 9, 117679-40-0; 10, 117679-41-1; 11, 117679-42-2; 12, 117679-43-3; 13, 117679-44-4; PhCH=CHCHO, 104-55-2; anilinoacetonitrile, 3009-97-0; allyl bromide, 106-95-6; crotonaldehyde, 4170-30-3; allylaniline, 589-09-3.

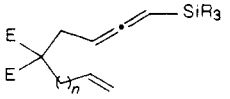
Synthesis of 4-(Dimethylphenylsilyl)buta-2,3-dien-1-ol and Its Use in Alkylation

Barry M. Trost* and James M. Tour

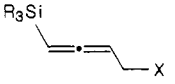
Department of Chemistry, Stanford University, Stanford, California 94305

Received July 15, 1988

During the course of our study directed toward the nickel–chromium-catalyzed cyclization of enallenes,[1] we became interested in using silyl-substituted enallenes such as 1. A logical synthesis of 1 involves the alkylation of



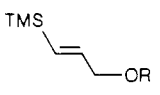
1
E = CO_2CH_3
R = alkyl or aryl



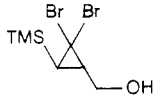
2
X = leaving group
R = alkyl or aryl

dimethyl malonate with the appropriate olefinic fragment and the allenylsilane 2. Although allenes are of widespread use as synthetic intermediates,[2] and there is a rapidly increasing number of isolated natural products containing an allene unit,[3] there are no reported syntheses of a (1,3-disubstituted-allenyl)silane with a potential leaving group in the 4-position. The allenylsilane 2 could have many applications as a bifunctional electrophile.

Initially, we attempted to synthesize 2 (X = OH, OTMS) by the addition of dibromocarbene to the alcohol 3[4] to form 4. The dibromide 4 could, upon addition of excess *n*-

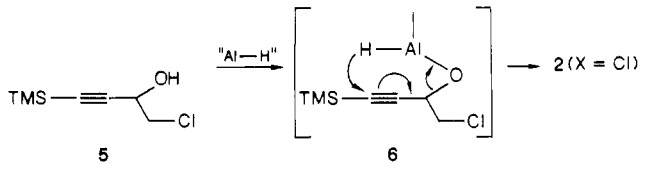


3a, R = H
b, R = TMS



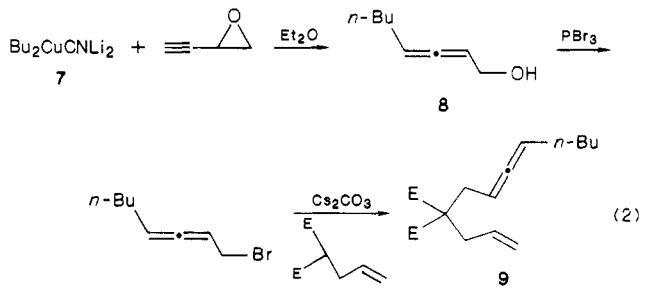
4

butyllithium,[5] form the desired allenylsilane 2 (X = OH). However, attempts to add dibromocarbene to **3a** or **3b** under phase-transfer conditions,[6] homogeneous basic conditions,[7] or with $PhHgCBr_3$[8] gave only trace amounts of the desired dibromide 4. Similarly, attempts to reduce the propargyl alcohol 5 with aluminum hydride reagents via six-membered transition state 6 were also unsuccessful (eq 1). We had observed that the higher order organo-



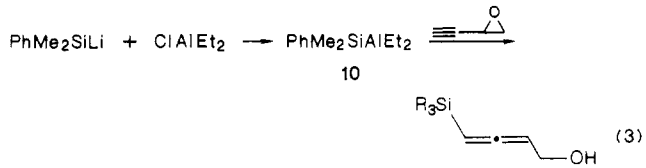
5 6 (1)

cuprate[9] 7 added cleanly to epoxybutyne to form the allenyl alcohol 8[10] in 58% yield.[11] Bromination of 8 with PBr_3 in 53% yield and cesium carbonate promoted alkylation with dimethyl allylmalonate in 97% yield afforded the butylated enallene 9 (eq 2). Attempts to follow an analogous procedure with the addition of $(PhMe_2Si)_2CuCNLi_2$[12] to epoxybutyne afforded only traces of the desired product 2 (X = OH).



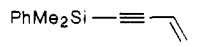
7 8 9 (2)

Conversely, we found that the silylalane 10,[13] easily generated from $PhMe_2SiLi$ and $ClAlEt_2$, added cleanly to epoxybutyne to form the desired allene 2 (X = OH) in 89% yield (eq 3). However, activation of the hydroxyl group



10 (3)

in 2 (X = OH) by conversion to the bromide using PBr_3 or to the tosylate using TsCl–pyridine or *n*-butyllithium and TsCl exclusively formed the enyne 11. Treatment



11

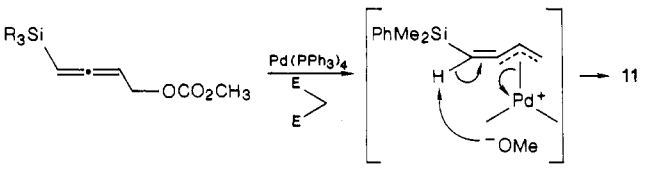
of 2 (X = OH) with CBr_4–Ph_3P[14] afforded a mixture of 2 (X = Br) and 11 in a 2:1 ratio, respectively. Attempted alkylation of this mixture with dimethyl allylmalonate and cesium carbonate yielded only the elimination product 11 and recovered dimethyl allylmalonate.

Since activation of the alcohol moiety on the allene 2 (X = OH) caused the system to become extremely prone to elimination, we turned our attention to palladium-catalyzed alkylation methodology.[15] Treatment of 2 (X

(1) Trost, B. M.; Tour, J. M. *J. Am. Chem. Soc.*, in press.
(2) Schuster, H. F.; Coppola, G. M. *Allenes in Organic Synthesis*, Wiley: New York, 1984. Rutledge, T. F. *Acetylenes and Allenes*, Reinhold: New York, 1969. Brandsma, L.; Verkruijsse, H. D. *Synthesis of Acetylenes, Allenes, and Cumulenes*; Elsevier: New York, 1981. Danheiser, R. L.; Carini, D. J.; Basak, A. *J. Am. Chem. Soc.* **1981**, *103*, 1604.
(3) Staudinger, H.; Ruzicka, L. *Helv. Chim. Acta* **1924**, *7*, 177. Clemer, W. D.; Solomans, I. A. *J. Am. Chem. Soc.* **1952**, *74*, 1870, 2245, 3838. Taylor, D. R. *Chem. Rev.* **1967**, *67*, 317. Bonnett, R.; Spark, A. A.; Tee, J. L.; Weedon, B. C. L. *Proc. Chem. Soc.* **1964**, 419. Sprecher, H. W.; Maier, R.; Barber, M.; Holman, R. T. *Biochemistry* **1965**, *4*, 1856. Suzuki, M.; Kurosawa, E. *Chem. Lett.* **1982**, 289. Warning, U.; Jakupovic, J.; Bohlmann, F.; Jones, S. P. *Ann.* **1987**, 467.
(4) Stork, G.; Jung, M. E.; Colvin, E.; Noel, Y. *J. Am. Chem. Soc.* **1974**, *96*, 3684.
(5) Maurin, R.; Bertrand, M. *Bull. Soc. Chim. Fr.* **1972**, 2349. Bertrand, M.; Maurin, M. *Bull. Soc. Chim. Fr.* **1967**, 2779. Bertrand, M.; Maurin, M.; Delépine, M. *C.R. Acad. Sci. Paris (C)* **1965**, *260*, 6122.
(6) Makosza, M.; Wawrzyniewicz, M. *Tetrahedron Lett.* **1969**, 4659.
(7) Skell, P. S.; Garner, A. Y. *J. Am. Chem. Soc.* **1956**, *78*, 5430.
(8) Seyferth, D.; Burlitch, J. M.; Minasz, R. J.; Mui, J. Y.-P.; Simmons, H. D., Jr.; Treiber, A. J. H.; Dowd, S. R. *J. Am. Chem. Soc.* **1965**, *87*, 4259.
(9) Lipshutz, B. H.; Wilhelm, R. S.; Kozlowski, J. A. *Tetrahedron* **1984**, *40*, 5005.
(10) Analogous 1-hydroxy-4-alkyl allenes have been prepared from epoxybutyne by using alkylboranes. See: Suzuki, A.; Miyaura, N.; Itoh, M. *Synthesis* **1973**, 305. 1-Hydroxy-4-alkynyl allenes have been prepared from epoxybutyne and organozinc reagents in the presence of Pd(0) catalysts. See: Kleijn, H.; Meijer, J.; Overbeek, G. C.; Vermeer, P. *Recueil* **1982**, *101*, 97.
(11) This reaction was unoptimized and the yield can undoubtedly be improved.
(12) Fleming, I.; Waterson, D. *J. Chem. Soc., Perkin Trans. 1* **1984**, 1809.
(13) The silylalane has been added to activated vinylcyclopropanes and vinylaziradines. See: Fugami, K.; Oshima, K.; Utimato, K.; Nozaki, H. *Bull. Chem. Soc. Jpn.* **1987**, *60*, 2509.
(14) Hooz, J.; Gilani, S. S. H. *Can. J. Chem.* **1968**, *46*, 86.

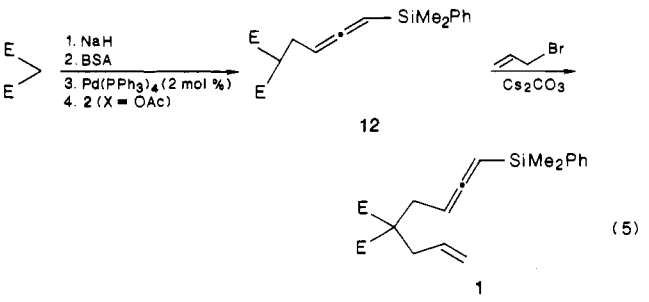
0022-3263/89/1954-0484$01.50/0

= OH) with *n*-butyllithium followed by methyl chloroformate afforded the carboxylate **2** (X = $OCOOCH_3$) in 90% yield. Alkylation with dimethyl malonate in the presence of $Pd(PPh_3)_4$ gave a mixture of the elimination product **11** and the alkylation product **12** in a 15:1 ratio, respectively. Apparently, under those conditions, methoxide promoted elimination of the (π-allyl)palladium species was faster than alkylation (eq 4). It is interesting



(4)

to note that deprotonation was faster than desilylation. Alternatively, analogous formation of the acetate **2** (X = OAc) proceeded in 97% yield. Treatment of sodium dimethyl malonate with bis(trimethylsilyl)acetamide (BSA) followed by addition of $Pd(PPh_3)_4$ and the allene acetate **2** (X = OAc) in THF at ambient temperature afforded the desired alkylated product **12** in 72% yield with <5% of the enyne **11** being formed. Treatment of **12** with allyl bromide and cesium carbonate gave the desired enallene **1** (n = 1) in 97% yield (eq 5).



(5)

In summary, the synthesis and alkylation of **2** was not attainable by several standard approaches. The sensitivity of **2** toward decomposition is highly evident by its rapid destruction when accompanied by the increased leaving group nature of X. Appropriate organometallic chemistry overcomes both problems and allows **2** to be employed as a readily available building block.

Experimental Section

General. All reactions were run under a positive pressure of nitrogen in flasks that were oven-dried (100 °C, 18 h) and allowed to cool under a stream of nitrogen. ^1H NMR spectra were recorded on a Nicolet NT300 at 300 MHz with chemical shifts reported in δ, parts per million (ppm), downfield from tetramethylsilane. Splitting patterns were designated as s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad. Coupling constants are reported in hertz. ^{13}C NMR spectra were recorded on a Varian XL400 spectrophotometer at 100 MHz and are reported in ppm relative to a center line of a triplet at 77.00 ppm which is attributed to the solvent. Infrared spectra were recorded on a Perkin-Elmer 1420 spectrophotometer as neat oils on sodium chloride plates. Tetrahydrofuran (THF) was distilled from sodium and benzophenone.

(Dimethylphenylsilyl)lithium.[13] To a flask containing THF (75 mL) was added at 0 °C with a strong back flow of nitrogen lithium wire (2.4 g, 350 mmol) [containing 1% sodium, hexane washed, pounded flat with a hammer, and cut with scissors just prior to addition]. To the suspension was rapidly added at 0 °C chlorodimethylphenylsilane (17.1 g, 17.0 mL, 100 mmol). The solution was stirred at 0 °C (cryostat) for 18 h. The solution was shown to be 1.0 M by titration[16] [*sec*-butyl alcohol (0.99 M in xylene), 1,10-phenanthrolene indicator] and used at this temperature for further experiments. The solution could be stored in the refrigerator (−3 °C) for 1 week.

4-(Dimethylphenylsilyl)buta-2,3-dien-1-ol (2, X = OH). To a solution of diethylaluminum chloride (0.84 g, 0.87 mL, 7.0 mmol) in THF (5 mL) was added at 0 °C (dimethylphenylsilyl)lithium (7.0 mL, 7.0 mmol, 1.0 M in THF) and the reaction mixture was stirred for 10 min at the same temperature. To the silylalane was added dropwise at −78 °C epoxybutyne[17] (0.32 g, 4.7 mmol) as a neat liquid. The reaction mixture was stirred at −78 °C for 1 h and allowed to warm to 0 °C for 1 h before being poured slowly into ice water. The two-phase solution was stirred rapidly for 10 min and filtered through a Celite plug to remove the aluminum salts. Ether was added and the organic layer was separated. The aqueous layer was extracted with ether (3×) and the combined organic layers were washed with brine and dried over magnesium sulfate. The volatiles were removed in vacuo and the crude material was distilled at 120–130 °C (0.2 mmHg, Kugelrohr) to afford 0.86 g (89%) of the title compound: IR (neat) 3350, 3070, 2960, 1943, 1432, 1254, 1118, 821 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.56 (m, 2 H), 7.39 (m, 3 H), 5.23 (dt, J = 6.0, 3.8 Hz, 1 H), 5.06 (q, J = 6.0 Hz, 1 H), 4.20 (m, 2 H), 1.12 (t, J = 7 Hz, 1 H), 0.40 (s, 3 H), 0.39 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.14, 133.58, 129.24, 127.79, 127.65, 84.57, 83.06, 60.66, −2.42; exact mass calcd for $C_{12}H_{16}OSi \cdot H_2O$ 186.0865, found 186.0860.

4-Acetoxy-1-(dimethylphenylsilyl)buta-1,2-diene (2, X = OAc). To a solution of 4-(dimethylphenylsilyl)buta-2,3-dien-1-ol (0.46 g, 2.26 mmol) in THF (4 mL) was added at −78 °C n-BuLi (1.9 mL, 2.4 mmol, 1.28 M in hexanes), and the solution was stirred for 5 min. Acetyl chloride (0.35 g, 0.32 mL, 4.5 mmol) was added in one portion and the solution was allowed to warm to room temperature for 15 min before being poured into a mixture of ice water and ether. The organic layer was separated and the aqueous layer was extracted with ether (2×). The combined organic phase was washed with saturated aqueous sodium bicarbonate and brine. The solution was dried over magnesium sulfate and the volatiles were removed in vacuo. The crude material was distilled at 130–140 °C (0.14 mmHg, Kugelrohr) to afford 0.54 g (97%) of the title compound: IR (neat) 3030, 2949, 1940, 1740, 1427, 1368, 1225, 1112, 816 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.52 (m, 2 H), 7.35 (m, 3 H), 5.22 (dt, J = 6.9, 2.5 Hz, 1 H), 5.01 (q, J = 6.9 Hz, 1 H), 4.55 (m, 2 H), 2.02 (s, 3 H), 0.38 (s, 6 H).

5,5-Dicarbomethoxy-1-(dimethylphenylsilyl)penta-1,2-diene (12). To a slurry of sodium hydride (72 mg, 1.5 mmol, 50% in oil) in THF (2 mL) was slowly added at 15 °C dimethyl malonate (0.20 g, 0.17 mL, 1.5 mmol). After the hydrogen evolution ceased (5 min), BSA (0.32 g, 0.40 mL, 1.6 mmol) was added at room temperature to the slurry and stirring was continued for 10 min. To the slurry was then added tetrakis(triphenylphosphine)palladium [prepared from tris(dibenzylideneacetone)dipalladium chloroform complex (10.3 mg, 0.010 mmol) and triphenylphosphine (26.2 mg, 0.10 mmol) in THF (1 mL)] and 4-acetoxy-1-(dimethylphenylsilyl)buta-1,2-diene (0.25 g, 1.0 mmol) in THF (1 mL). After being stirred for 30 min, the solution was poured into a mixture of ether and water. The organic layer was separated and the aqueous phase was extracted with ether (2×). The combined organic phase was washed with brine and dried over magnesium sulfate. The volatiles were removed in vacuo and the crude material was purified by column chromatography [silica gel, hexane/ether (7/1)] to afford a 0.23 g (72%) of the title compound: IR (neat) 2944, 1942, 1735, 1437, 1249, 1112, 818 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.52 (m, 2 H), 7.34 (m, 3 H), 5.10 (dt, J = 7.0, 3.5 Hz, 1 H), 4.82 (q, J = 7.0 Hz, 1 H), 3.70 (s, 3 H), 3.69 (s, 3 H), 3.40 (t, J = 8 Hz, 1 H), 2.57 (dd, J = 7.1, 3.5 Hz, 1 H), 2.54 (dd, J = 7.2, 3.5 Hz, 1 H), 0.35 (s, 3 H), 0.34 (s, 3 H); exact mass calcd for $C_{17}H_{22}O_4Si$ 318.1288, found 318.1281.

5,5-Dicarbomethoxy-1-(dimethylphenylsilyl)octa-1,2,7-triene (1, n = 1). To a solution of 5,5-dicarbomethoxy-1-(dimethylphenylsilyl)penta-1,2-diene (0.22 g, 0.69 mmol) in acetone

(15) Trost, B. M. *Acc. Chem. Res.* **1980**, *13*, 385. Ahmar, M.; Cazes, B.; Gore, J. *Tetrahedron* **1987**, *43*, 3453. Djahanbini, D.; Cazes, B.; Gore, J. *Tetrahedron* **1987**, *43*, 3441.

(16) Watson, S. C.; Eastham, J. F. *J. Organomet. Chem.* **1967**, *9*, 165.

(17) Lespieau, R. *Bull. Soc. Chim. Fr.* **1928**, *43*, 199. Gross, H. J. *J. Prakt. Chem.* **1963**, *21*, 99.

(3 mL) [freshly distilled over sodium sulfate and BSA] were added at room temperature allyl bromide (0.24 g, 0.17 mL, 2.0 mmol) and cesium carbonate (0.46 g, 1.4 mmol). The solution was heated to reflux for 3 h, cooled, and poured into a mixture of ice water and ether. The organic phase was separated and the aqueous phase was extracted with ether (3×). The combined organic phase was washed with brine and dried over magnesium sulfate. The solvent was removed in vacuo and the crude material was purified by column chromatography [silica gel, hexane/ether (6/1)] to afford 0.24 g (97%) of the title compound: IR (neat) 2944, 1942, 1736, 1640, 1433, 1215, 1112, 818 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.51 (m, 2 H), 7.35 (m, 3 H), 5.62 (ddt, J = 17, 10, 6 Hz, 1 H), 5.08 (d, J = 17 Hz, 1 H), 5.05 (m, 2 H), 4.63 (q, J = 7.5 Hz, 1 H), 3.68 (s, 6 H), 2.66 (d, J = 7.5 Hz, 2 H), 2.60 (dd, J = 7.5, 3 Hz, 2 H), 0.33 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 211.68, 171.14, 171.07, 138.18, 133.60, 132.29, 129.17, 127.80, 119.21, 80.98, 77.67, 57.97, 52.39, 52.33, 36.70, 31.54, -2.36; exact mass calcd for $C_{20}H_{26}O_4Si$ 358.1601, found 358.1597.

Acknowledgment. We thank the National Science Foundation and the National Institutes of Health for their generous support of our programs. J.M.T. thanks the National Institutes of Health for a postdoctoral fellowship for a portion of his stay in these laboratories. Mass spectra were gratefully provided by the Mass Spectrometry Facility, University of California–San Francisco, supported by the NIH Division of Research Resources.

Registry No. 1 (n = 1), 117687-47-5; 2 (X = OH), 117687-48-6; 11, 18042-55-2; 12, 117687-50-0; BSA, 10416-59-8; $PhMe_2SiLi$, 3839-31-4; $ClAlEt_2$, 96-10-6; $PhMe_2SiCl$, 768-33-2; $PhMe_2SiCH{=}C{=}CHCH_2OCO_2CH_3$, 117687-49-7; $MeO_2CCH_2CO_2Me$, 108-59-8; allyl bromide, 106-95-6; epoxybutyne, 6924-81-8.

Studies on Benzylchlorocarbene

Michael T. H. Liu* and Raghava V. Suresh

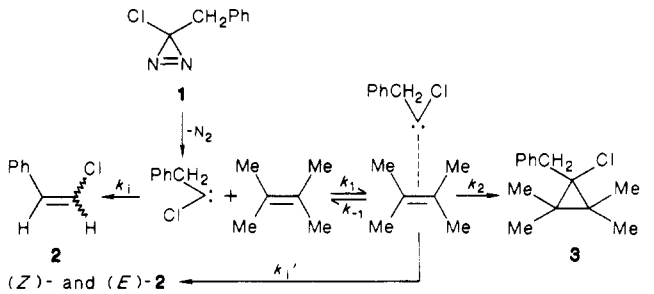
Department of Chemistry, University of Prince Edward Island, Charlottetown, P.E.I., Canada C1A 4P3

Received March 16, 1988

Photolysis or thermolysis of 3-chloro-3-benzyldiazirine (1) undergoes dinitrogen extrusion and in the presence of alkene, cyclopropanation directly competes with the intramolecular 1,2-hydrogen migration.[1,2] The intervention of a reversibly formed carbene–alkene complex, based on the Turro–Moss model,[3] has been advanced to rationalize the kinetic data for competitive carbene reactions.

Laser flash photolysis (LFP) indicates that increasing solvent polarity has no significant influence on the rates of carbene–alkene cycloaddition.[3,4] Here we report the results of our investigation on the solvent-induced effect of such a carbenic process, which involves a competing intramolecular rearrangement. Also, though it is well known[5,6] that 1,2-H migration in a carbene results in a mixture of isomeric olefins, the energetics of the equilibrium between the carbene conformers is hitherto not known. Here we determine if the isomeric olefins produced from the thermal decomposition of diazirine 1 are in thermodynamic quantity.

Scheme I



Results and Discussion

3-Chloro-3-benzyldiazirine (1) was synthesized by Graham's method[7] and 0.02 M solutions of 1 in the presence of excess tetramethylethylene (TME) in acetonitrile were photolyzed and thermolyzed over the 20–95 °C temperature range. The cyclopropane 3/chlorostyrene 2 product ratios as well as the Z/E ratios of 2 are given in Table I. A mechanistic model (Scheme I) accounts not only for the curvature in 3/2 vs [TME] plots but also predicts the change in Z/E ratio of 2 with increasing TME concentration. Application of steady-state treatment to the scheme leads to eq 1. Values for k_i/k_t were obtained by

$$\frac{2}{3} = \frac{k_i}{k_t} \times \frac{1}{[\mathrm{TME}]} + \frac{k_i'}{k_2} \qquad (1)$$

correlating 2/3 vs 1/[TME] where k_t is equal to k_1k_2/k_{-1}. Plots of log k_i/k_t vs $1/T$ for thermolysis and photolysis data gave a single Arrhenius line with $E_i - E_t = 4.6 \pm 0.2$ kcal mol^{-1} and $A_i/A_t = 10^{3.6\pm0.1}$.

Absolute rate constants for the reactions of phenylchlorocarbene with TME in isooctane, acetonitrile, and toluene at room temperature are 2.8×10^8, 1.1×10^8, and 1.4×10^8 M^{-1} s^{-1}, respectively.[3] Additionally, the absolute rate constants for the reactions of phenylchlorocarbene with 1-hexene, diethyl fumarate, and ethyl acrylate in toluene or ethyl acetate are similar,[4] indicating that the cycloadditions are not sensitive to solvent polarity and hence no significant change in the E_t. If E_t is taken[8] to be -1.7 kcal mol^{-1} and $A_t = 1.9 \times 10^7$ M^{-1} s^{-1}, the kinetic parameters for 1,2-H shift for benzylchlorocarbene in CH_3CN can be calculated, hence, $k_i(CH_3CN) = 10^{11.0}$ exp $-(2900$ cal/mol/$RT)s^{-1}$. In an earlier paper,[2] we reported k_i(isooctane) $= 10^{12.2}$ exp $-(6400$ cal/mol/$RT)s^{-1}$, which predicts a life-time for $C_6H_5CH_2\ddot{C}Cl$ in the order of 40 ns at room temperature, which is in excellent agreement with LFP[9] data of 30 ns. Recently, it was shown[1] that 1,2-H shift in Bz$\ddot{C}$Cl involves a charge development in the transition state. In the present study, the lowering of activation energy by ~3 kcal mol^{-1} for 1,2-H shift of Bz$\ddot{C}$Cl in CH_3CN as compared to isooctane can be explained in terms of the increased stability of such a charge-developed transition state by CH_3CN. While our finding is in broad agreement with the work of Tomioka et al.[10] the marked

(1) Liu, M. T. H.; Soundararajan, N.; Paike, N.; Subramanian, R. *J. Org. Chem.* **1987**, *52*, 4223.
(2) Liu, M. T. H. *J. Chem. Soc., Chem. Commun.* **1985**, 982. Since E_a = 6.4 kcal mol^{-1} and $A = 1.5 \times 10^{12}$ s^{-1}, the overall $\Delta G^{\ddagger}$ for 1,2-H migration in benzylchlorocarbene at 400 K is 7.7 kcal mol^{-1}. $\Delta G^{\ddagger} = \Delta H^{\ddagger} - T\Delta S^{\ddagger}$ and $E_a = \Delta H^{\ddagger} + RT$.
(3) Gould, I. R.; Turro, N. J.; Butcher, J., Jr.; Doubleday, C., Jr.; Hacker, N. P.; Lehr, G. F.; Moss, R. A.; Cox, D. P.; Guo, W.; Munjal, R. C.; Perez, L. A.; Fedorynski, M. *Tetrahedron* **1985**, *41*, 1587.
(4) Soundararajan, N.; Platz, M. S.; Jackson, J. E.; Doyle, M. P.; Oon, S.-M.; Liu M. T. H.; Anand, S. M. *J. Am. Chem. Soc.* **1988**, *110*, 7143.
(5) Jones, W. M. *Rearrangements in Ground and Excited States*; de Mayo, P., Ed.; Academic Press: New York, 1980; Vol. 1, p 95.
(6) Doyle, M. P. In *Chemistry of Diazirines*; Liu, M. T. H., Ed.; CRC: Boca Raton, FL, 1987; Chapter 8.
(7) Graham, W. H. *J. Am. Chem. Soc.* **1965**, *87*, 4396.
(8) Turro, N. J.; Lehr, G. F.; Butcher, G. F.; Moss, R. A.; Guo, W. *J. Am. Chem. Soc.* **1982**, *104*, 1754.
(9) Jackson, J. E.; Soundararajan, N.; Platz, M. S.; Liu, M. T. H. *J. Am. Chem. Soc.* **1988**, *110*, 5595 and unpublished results.
(10) Tomioka, H.; Hayashi, N.; Izawa, Y. *Chem. Lett.* **1986**, 695.

0022-3263/89/1954-0486$01.50/0