Solvolysis of [3.3.2]Propellan-9-yl Tosylate

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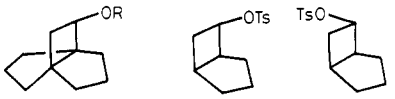
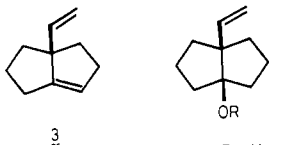
It is well-known that the solvolysis of cyclobutyl derivatives proceeds with σ participation of the cyclobutane bond and that the ring opening of the cyclobutane bond occurs so as to ensure maximum overlap of the ring bond orbital and the developing p orbital, and, therefore, the solvolysis reactivity is sensitive to the conformation of the cyclobutane ring.[1] In this respect, the solvolysis of many types of polycyclic cyclobutane derivatives has been studied.[1,2] We have been interested in the carbonium ion rearrangements of the propellanes involving one or two cyclobutane rings, [*m.n.*2]propellanes ($m \geq 3$, $n \geq 2$), with a view to synthesizing important polycarbocyclic ring systems.[3] In this connection, we report here the solvolysis of [3.3.2]propellan-9-yl tosylate (**1d**), a tricyclic cyclobutane



1a R = H 2a 2b
1b R = Ac
1c R = COH
1d R = Ts

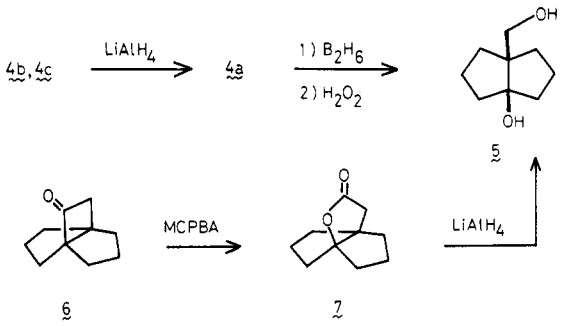
derivative having a propellane framework. The constituent bicyclic tosylates, *endo*- and *exo*-bicyclo[3.2.0]hept-6-yl tosylates (**2a,b**), have been shown to undergo solvolysis with ring opening of the internal (1,5-bond) and the external (1,7-bond) cyclobutane bonds, respectively.[4] Since the tosylate **1d** may be regarded to have the structural features of both endo tosylate **2a** and exo tosylate **2b**, σ participation of both internal and external bonds of the cyclobutane ring would be expected.

1d was prepared from the propellanol **1a** which was reported previously.[5] The products obtained from the buffered solvolysis are summarized in Table I. As shown in Table I, the buffered acetolysis of **1d** gave the diene **3**



3 4a R = H
4b R = Ac
4c R = COH

Scheme I



as the major product along with the unrearranged acetate **1b**, and, in the buffered formolysis, the cis formate **4c** was obtained together with the unrearranged ester **1c**. The structures of the unrearranged esters **1b** and **1c** were confirmed by their lithium aluminum hydride conversion into the alcohol **1a**. The cis stereochemistry of **4c** as well as that of the acetate **4b**, described later, was established by the following reactions (shown in Scheme I): the lithium aluminum hydride reduction of both **4b** and **4c** gave the alcohol **4a**. The hydroboration–oxidation of **4a** afforded the diol which was identical (IR, GLC, and melting point) with the cis diol **5** derived from the cyclobutanone **6**[5] by Baeyer–Villiger oxidation to yield the γ-lactone **7**, and subsequent lithium aluminum hydride reduction of **7**. It is of significance that, in the buffered solvolysis of **1d**, the products derived from the external bond opening were always obtained without any formation of the internal bond-cleavage products.

The rates of the buffered acetolysis of **1d** are listed in Table II together with the rate data of **2a** and **2b**. As can be seen from Table II, **1d** undergoes solvolysis at a rate slightly greater than that of **2b** but considerably smaller than that of **2a**. The kinetic results as well as the product study described above indicate that the ionization of **1d** occurs with σ participation of the external cyclobutane bond but with little participation of the internal bond. On the basis of the dihedral angles between the methine and the methylene protons of the cyclobutane ring (approximately 30° and 160°) calculated from the vicinal coupling constants of the methine proton in the ^{1}H NMR spectrum of **1d** (dd, J = 6, 8 Hz) by using the Karplus equations,[6] it is suggested that the cyclobutane ring of **1d** has a considerably puckered conformation despite the rigidity of [3.3.2]propellane framework. Therefore, it may be reasonable to consider that the assistance of the external bond in the ionization is favorable because of the nearly antiperiplanar arrangement between the leaving group and the external cyclobutane bond. However, σ participation of the internal bond may be kinetically inhibited by the strained nature of the transition state leading to the carbonium ions such as bicyclo[3.3.2]dec-1(9)-en-5-yl and/or tricyclo[3.3.2.0^{1,10}]decan-5-yl cations.[7]

In connection with our interest in the transformation of [*n*.3.2]propellanes ($n \geq 3$) into thermodynamically more stable carbocyclic skeletons by multiple-step carbonium ion rearrangements,[2e] the unbuffered solvolysis of **1d** was also examined (Table I). As shown in Table I, the initial products obtained from the unbuffered acetolysis were the diene **3** and the acetate **1b**, as in the case of the buffered acetolysis. Although the acetate **4b**, formed by the acid-

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(6) Karplus, M. *J. Chem. Phys.* **1959**, *30*, 11.

(7) The formation of highly strained allylcarbinyl- and cyclopropylcarbinyl-type bridgehead cations, however, was observed in the cases of the carbonium ion rearrangement of more strained [*n*.2.2]propellanes (n = 3–6).[3b,f,g]

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Table I. Product Distribution from the Solvolysis of 1d

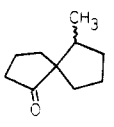
conditions	combined yield of products, %	product, % 3	1b	4b	1c	4c	8	9b	9c
HOAc with 2 equiv of NaOAc, 60 °C, 50 h	95	69	31						
HOAc, 60 °C, 1 h		88	12						
HOAc, 60 °C, 50 h	87	18	12	70					
HO_2CH with 2 equiv of NaO_2CH, 60 °C, 24 h	98				33	67			
HO_2CH, 60 °C, 1 h						80	5	8	7
HO_2CH, 60 °C, 24 h	64						48	31	22

Table II. Kinetic Data for the Buffered Acetolysis of 1d

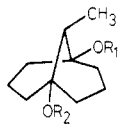
compd	temp, °C	k, s^{-1}	$\Delta H^{\ddagger}$, kcal/mol	$\Delta S^{\ddagger}$, eu	k_{rel}
1d	70.0	$(1.92 \pm 0.01) \times 10^{-4}$			
	60.0	$(6.07 \pm 0.03) \times 10^{-5}$			
	50.0	$(1.79 \pm 0.01) \times 10^{-5}$	25.5	−1.6	6.9
2a[a]	50	1.2×10^{-3}			460
2b[a]	50	2.6×10^{-6}			1.0

[a] Reference 4.

catalyzed addition of acetic acid to the diene **3**, was obtained on prolonged reaction, no skeletal rearrangement was observed. On the other hand, in the case of the unbuffered formolysis, the acid-catalyzed rearrangement of the initial product **4c** took place to give 1-methylspiro[4.4]nonan-6-one (**8**), the diformate ester (**9b**) of 9-methylbicyclo[3.3.1]nonane-1,5-diol (**9a**), and the monoformate (**9c**) of **9a**. The structures of **8**, **9b**, and **9c** were



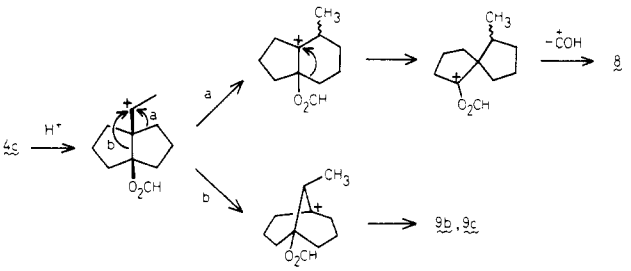
8



9a $R_1 = R_2 = H$
9b $R_1 = R_2 = COH$
9c $R_1 = H$, $R_2 = COH$

elucidated on the basis of spectroscopic as well as mechanistic considerations as described below: the mass spectrum and the elemental analysis of **8** indicate that **8** has a molecular formula of $C_{10}H_{16}O$. The carbonyl absorption in the IR spectrum was at 1720 cm^{-1}, characteristic of cyclopentanone. The presence of a methyl group on a tertiary carbon was suggested by the doublet at δ 0.82 in the ^1H NMR spectrum and the quartet at δ 15.5 as well as the doublet at δ 40.2 in the ^{13}C NMR spectrum. Finally, the singlet absorption at δ 59.3 in the ^{13}C NMR spectrum confirmed the spiro[4.4]nonane skeleton of **8**. The symmetric diester **9b** showed only one formyl proton at δ 7.82 and a methyl proton doublet at δ 1.01 in the ^1H NMR spectrum. The salient feature of the ^{13}C NMR spectrum included two singlets at δ 160.0 and 86.5, a doublet at δ 44.4, and a highly shielded methyl quartet at δ 7.9. Because of its instability, the structure of the hydroxy ester **9c** was confirmed by lithium aluminum hydride reduction into the crystalline diol **9a** which was prepared from **9b** in the same manner. In view of the results on the solvolytic rearrangement of the bicyclo[3.3.0]oct-1-yl carbinyl cation[8] and on the pinacol rearrangement of bicyclo[4.3.0]nonane-1,6-diols,[9] it may be reasonable to postulate the mechanistic pathway from **4c** to **8** and **9b** as shown in Scheme II.[10]

Scheme II



Experimental Section[11]

Tosylate (1d). Compound **1d** was prepared from **1a**[5] in the usual manner followed by an aqueous workup in 87% yield: mp 35–36 °C; IR 1590, 1350, 1175, 1160, 645, 545 cm^{-1}; ^1H NMR (CCl_4) δ 1.00–2.25 (m, 14 H), 2.44 (s, 3 H), 4.44 (dd, J = 6, 8 Hz, 1 H), 7.24 (d, 2 H), 7.68 (d, 2 H). Anal. Calcd for $C_{17}H_{22}O_3S$: C, 66.64; H, 7.24; S, 10.46. Found: C, 66.36; H, 7.11; S, 10.22.

Preparative Solvolysis of 1d. For the preparative solvolysis, a 0.05 M solution of **1d** in acetic acid or formic acid was heated at 60 °C for 50 or 24 h. The solution was diluted with water and extracted with ether. The extracts were washed with sodium bicarbonate solution and water and then dried over anhydrous sodium sulfate (Na_2SO_4). After evaporation of the solvent, the products were analyzed by GLC and separated by preparative GLC. In the case of the unbuffered formolysis, the products were separated by chromatography on silica gel prior to purification by GLC. The results are summarized in Table I.

1b: IR 1730, 1225, 1035 cm^{-1}; mass spectrum, m/e 194 (M^+, trace), 108 (base); ^1H NMR (CCl_4) δ 1.00–2.28 (m, 17 H, s at δ 1.98), 4.55 (dd, J = 6, 8 Hz, 1 H). Anal. Calcd for $C_{12}H_{18}O_2$: C, 74.19; H, 9.34. Found: C, 74.24; H, 9.41.

1c: IR 1710, 1160 cm^{-1}; mass spectrum, m/e 180 (M^+), 134 ($M^+ - HO_2CH$), 108 (base); ^1H NMR (CCl_4) δ 1.00–2.40 (m, 14 H), 4.68 (dd, J = 6, 8 Hz, 1 H), 7.90 (s, 1 H). Anal. Calcd for $C_{11}H_{16}O_2$: C, 73.30; H, 8.95. Found: C, 73.31; H, 8.63.

3: IR 3040, 1625, 985, 900 cm^{-1}; mass spectrum, m/e 134 (M^+); ^1H NMR (CCl_4) δ 1.10–2.84 (m, 10 H), 4.87 (2 d, J = 10, 17 Hz, 2 H), 5.20–5.36 (m, 1 H), 5.76 (dd, J = 10, 17 Hz, 1 H). Anal. Calcd for $C_{10}H_{14}$: C, 89.49; H, 10.51. Found: C, 89.27; H, 10.66.

(8) Flath, R. A. Ph.D. Thesis, University of California, Berkeley, CA, 1963. Cited in: Dauben, W. G.; Chitwood, J. L. *J. Am. Chem. Soc.* **1970**, *92*, 1624.
(9) Mundy, B. P.; Otzenberger, R. D. *J. Org. Chem.* **1973**, *33*, 2109.
(10) Whether the hydroxy ester **6c** was formed by the solvolysis or during the workup procedure is uncertain at present.
(11) Melting points are uncorrected. IR spectra were recorded with a JASCO IR-G spectrometer. ^1H and ^{13}C NMR spectra were obtained on a JEOL JNM-PS-100 and JEOL JNM-FX-60S spectrometers, respectively, with tetramethylsilane as an internal standard. Mass spectra were measured with a Hitachi RMU-6E spectrometer. Analytical GLC was carried out on a Hitachi 163 gas chromatograph, and preparative GLC separation was conducted on a Varian Aerograph 920 chromatograph with 10% FFAP and 5% SE-30 columns.

4b: IR 3050, 1720, 1240, 1010, 890 cm^{-1}; mass spectrum, *m/e* 194 (M^+), 152 (base), 134 (M^+ – HOAc); ^1H NMR (CCl_4) δ 1.08–2.40 (m, 15 H, s at δ 1.89), 4.92 (2 d, *J* = 11, 17 Hz, 2 H), 5.96 (dd, *J* = 11, 17 Hz, 1 H). Anal. Calcd for $C_{12}H_{18}O_2$: C, 74.19; H, 9.34. Found: C, 73.84; H, 9.18.

4c: IR 3050, 1705, 1160, 985, 895 cm^{-1}; mass spectrum, *m/e* 180 (M^+, trace), 134 ($M^+ - HO_2CH$), 91 (base); ^1H NMR (CCl_4) δ 1.40–2.40 (m, 12 H), 4.93 (2 d, *J* = 11, 17 Hz, 2 H), 5.95 (dd, *J* = 11, 17 Hz, 1 H), 7.80 (s, 1 H). Anal. Calcd for $C_{11}H_{16}O_2$: C, 73.30; H, 8.95. Found: C, 73.00; H, 8.84.

8: IR 1720, 1145 cm^{-1}; mass spectrum, *m/e* 152 (M^+), 97 (base); ^1H NMR (CCl_4) δ 0.82 (d, *J* = 6 Hz, 3 H), 1.04–2.44 (m, 13 H); ^{13}C NMR ($CDCl_3$) δ 224.0 (s), 59.3 (s), 40.2 (d), 38.1 (t), 37.5 (t), 33.7 (t), 30.9 (t), 22.9 (t), 19.4 (t), 15.5 (q). Anal. Calcd for $C_{10}H_{16}O$: C, 78.89; H, 10.59. Found: C, 78.61; H, 10.72.

9b: mp 32–33 °C; IR 1705, 1160, 890 cm^{-1}; mass spectrum, *m/e* 226 (M^+, not detected), 180 ($M^+ - HO_2CH$), 134 (base); ^1H NMR ($CDCl_3$) δ 1.01 (d, *J* = 6 Hz, 3 H), 1.50–2.80 (m, 13 H), 7.82 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 160.0 (s, 2 C), 86.5 (s, 2 C), 44.4 (d), 35.4 (t, 2 C), 28.5 (t, 2 C), 20.6 (t), 19.9 (t), 7.9 (q). Anal. Calcd for $C_{12}H_{18}O_4$: C, 63.70; H, 8.02. Found: C, 63.86; H, 8.06.

9c: IR 3400, 1700, 1170, 980 cm^{-1}; mass spectrum, *m/e* 198 (M^+, not detected), 152 ($M^+ - HO_2CH$), 124 (base); ^1H NMR (CCl_4) δ 0.98 (d, *J* = 6 Hz, 3 H), 1.36–2.60 (m, 14 H), 7.87 (s, 1 H).

Kinetic Measurements. The rates of the buffered acetolysis of **1d** were measured by the titrimetric method as previously described.[3c]

Lithium Aluminum Hydride Reduction of 1b,c and 4b,c. A 60-mg (0.31 mmol) sample of **1b** in 2 mL of ether was added dropwise to a suspension of 11.8 mg (0.31 mmol) of lithium aluminum hydride in 2 mL of ether, and the mixture was stirred at room temperature for 1 h. Water was added followed by 1 N hydrochloric acid, and the organic layer was separated. The aqueous layer was extracted with ether, and the combined extracts were washed with sodium bicarbonate solution and water and then dried (Na_2SO_4). Evaporation of the solvent gave 44 mg (93%) of **1a**.[5] In a similar manner, 26 mg of **1c** gave 20 mg (95%) of **1a**, 20 mg of **4b** gave 14 mg (93%) of **4a**, and 55 mg of **4c** gave 46 mg (98%) of **4a**.

4a: IR 3400, 3050, 1625, 1110, 990, 890 cm^{-1}; mass spectrum, *m/e* 152 (M^+); ^1H NMR (CCl_4) δ 1.00–2.40 (m, 13 H), 5.02 (2 d, *J* = 11, 17 Hz, 2 H), 5.85 (dd, *J* = 11, 17 Hz, 1 H). Anal. Calcd for $C_{10}H_{16}O$: C, 78.89; H, 10.59. Found: C, 78.59; H, 10.66.

Hydroboration–Oxidation of 4a. To a stirred suspension of 46 mg (0.63 mmol) of **4a** and 18 mg (0.47 mmol) of sodium borohydride in 5 mL of THF was added 0.08 mL (0.63 mmol) of boron trifluoride etherate dropwise via syringe, and the mixture was stirred at room temperature for 3 h. Water (0.05 mL), 0.1 mL of 3 N sodium hydroxide solution, and 0.1 mL of 30% hydrogen peroxide were added dropwise, and then the solution was allowed to stand overnight. The organic layer was separated, and the aqueous layer was extracted with ether. The combined organic layer was washed with saturated sodium chloride solution and dried (Na_2SO_4). Evaporation of the solvent gave 95 mg (89%) of the diol **5** as white solid which was recrystallized from ether–petroleum ether: mp 99–100 °C; IR 3250, 1030, 1000 cm^{-1}; mass spectrum, *m/e* 170 (M^+, trace), 128 (base); ^1H NMR ($CDCl_3$) δ 1.30–2.00 (m, 14 H), 2.60–3.40 (br, 2 H), 3.79 (t, 2 H). Anal. Calcd for $C_{10}H_{18}O_2$: C, 70.54; H, 10.66. Found: C, 70.79; H, 10.64.

Alternate Preparation of 4a. A 2.46-g (10 mmol) sample of 70% *m*-chloroperbenzoic acid (MCPBA) was added portionwise to a solution of 980 mg (6.5 mmol) of the cyclobutanone **6** in 30 mL of chloroform, and the solution was stirred at room temperature for 20 h. The solution was washed with sodium sulfite solution, sodium bicarbonate solution, and water and then dried (Na_2SO_4). After evaporation of the solvent, the residue was chromatographed on silica gel. Elution with 20% ether–petroleum ether gave 494 mg (48%) of the γ-lactone **7**: IR 1760, 1230, 1190, 1160, 1130, 950 cm^{-1}; mass spectrum, *m/e* 166 (M^+), 110 (base); ^1H NMR (CCl_4) δ 1.40–2.30 (m, 12 H), 2.43 (s, 2 H).

A 431-mg sample of **7** was reduced with lithium aluminum hydride in a manner similar to that described for **1b** to afford 336 mg (87%) of the diol **5** which was identical in melting point, IR, and GLC with the sample obtained by the hydroboration–oxidation of **4a**.

Lithium Aluminum Hydride Reduction of 9b and 9c. The reduction of **9b** and **9c** was carried out as described for that of **1b** except that chloroform was used for extraction. The diol **9a** was obtained from **9b** and **9c** in 67% and 47% yields, respectively. A pure sample of **9a** was obtained by recrystallization from ether: mp 148–150 °C; IR 3300, 980 cm^{-1}; mass spectrum, *m/e* 170 (M^+, trace), 152 (base, $M^+ - H_2O$); ^1H NMR ($CDCl_3$) δ 1.03 (d, *J* = 6 Hz, 3 H), 1.20–2.20 (m, 15 H). Anal. Calcd for $C_{10}H_{18}O_2$: C, 70.54; H, 10.66. Found: C, 70.15; H, 10.73.

Registry No. **1a**, 79483-08-2; **1b**, 79483-09-3; **1c**, 79483-10-6; **1d**, 79483-11-7; **3**, 79483-12-8; **4a**, 79483-13-9; **4b**, 79483-14-0; **4c**, 79483-15-1; **5**, 79483-16-2; **6**, 71734-13-9; **7**, 79483-17-3; **8**, 79547-85-6; **9a**, 79483-18-4; **9b**, 79483-19-5; **9c**, 79483-20-8.

Simple and Direct Synthesis of *trans*-1,2-Bis(tri-*n*-butylstannyl)ethylene

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Vinylstannanes are versatile precursors to a variety of funtionally substituted organic molecules. While indirect methods of synthesis from a number of sources (alkynes,[2] alkynoates,[3] vinylmetals,[4] β-substituted acrylates[6]) exist, delivery of an intact vinylstannyl residue to a substrate may be readily accomplished with *trans*-1,2-bis(tri-*n*-butylstannyl)ethylene (**3**) via transmetalation with *n*-butyllithium[7] or electrophilic destannylation.[8] Although **3** is available in excellent yield by hydrostannation of tri-*n*-butylethynylstannane (**1**) with tri-*n*-butyltin hydride,[9] the large-scale preparation of **1** is often attended with difficulty. This problem prompted the Corey group to devise a more complicated procedure utilizing derivatives of chloroacetylene.[10]

Herein we describe an efficient synthesis of **1** from a solution of tri-*n*-butyltin chloride and lithium acetylide in tetrahydrofuran which affords the prduct directly in 75% yield (Scheme I). The remaining 25% of the reaction mixture consists largely of bis(tri-*n*-butylstannyl)acetylene (**2**) which may be conveniently separated from **1** by simple distillation. During the course of this study, a procedure was developed for converting the side product **2** to **1** in 65% yield by treatment with lithium acetylide in tetrahydrofuran. Thus, large quantities of **1** are readily available in yields in excess of 90%.

Experimental Section

Tri-*n*-butylethynylstannane (1). A solution of 150 mL (233 mmol) of *n*-butyllithium in hexane in 500 mL of dry tetrahydrofuran was stirred under an acetylene atmosphere at 0 °C

(1) (a) Department of Medicinal Chemistry. (b) Department of Chemistry. (c) This research was supported by the National Institutes of Health Grant CA 19898 and the Department of Energy Contract DEACO2-76EYO4115.

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