Solvolysis of the Pentacyclo[4.4.0.0^{2,4}.0^{3,8}.0^{5,7}]decane System[1]

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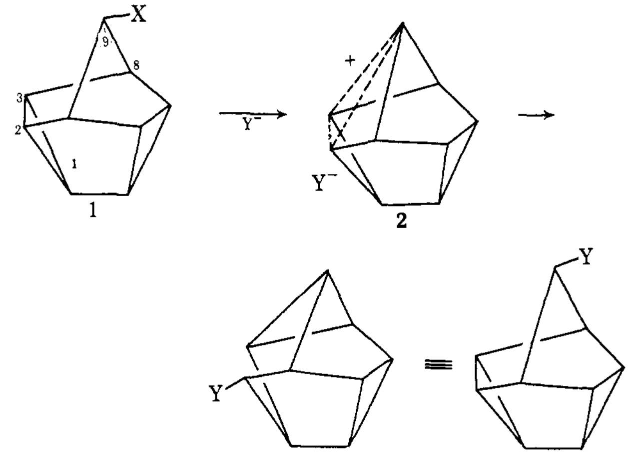
Abstract: The pentacyclo[4.4.0.0^{2,4}.0^{3,8}.0^{5,7}]decane 9-tosylate (**5**) solvolyzes in acetic acid with a rate of $k = 7.02 \times 10^{-5}$ sec^{-1} at 25.0°. The carbonyl stretching frequency of the corresponding ketone, according to the Foote correlation, would predict a rate slightly slower than cyclohexyl tosylate while the actual rate is over 10^3 faster. This suggests that homocyclopropyl participation in the transition state is increasing the rate over that value expected from just strain release. The tosylate **5** yielded 97% of one acetate upon solvolysis and the chemical and spectral data indicate the product to be 2-acetoxytetracyclo[5.3.0.0.^{3,5}.0^{4,8}]dec-9-ene (**6**).

The carbonium ion produced by the solvolysis of the pentacyclo[4.3.0.0^{2,4}.0^{3,8}.0^{5,7}]nonyl system **1**[3] is capable of degeneracy by means of a homocyclopropylcarbinyl rearrangement **1** → **2** → **1′**. For example, capture by solvent at the position indicated in **2** will result in the same ring system. Evidence for such a threefold symmetry has been gained by means of deuterium labeling at the 9 position of **1**; after solvolysis the deuterium was equally distributed between carbons 2, 3, and 9. The rate of solvolysis of **1** (X = *p*-nitrobenzoate) is 10^{12} times faster than that of a similar derivative in the 7-norbornyl system.[3,4] This large rate enhancement has been attributed to the extremely favorable geometry for homocyclopropyl participation in the solvolytic transition state **2**, a proposal similar to that advanced for the structurally related *endo*-*anti*-8-tricyclo[3.2.1.0^{2,4}]octyl *p*-nitrobenzoate (**3**).[5]



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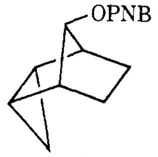
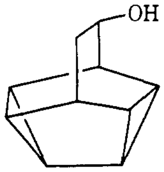


3

In this homocyclopropyl participation both the steric relationship of the edge-on participation of the cyclopropane ring and the symmetry (and in the limiting case, degeneracy) of the resulting ion can be of importance. In the recently prepared pentacyclo[4.4.0.0^{2,4}.0^{3,8}.0^{5,7}]decyl system **4**,[6] the cyclopropane rings are also in a position to donate electron density to a positive center at position 9, but the resulting ion cannot rearrange to equivalent structures. Thus, a study of this ring system should give information pertaining to these two aspects of homocyclopropyl participation. The starting alcohol **4** was prepared as described earlier,[6] and the crystalline tosylate **5** was obtained in the usual manner. Reduction of **5** with lithium in ammonia resulted in recovery of the unrearranged alcohol **4**.



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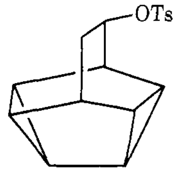

5

Preparative Acetolysis of Tosylate 5. When the tosylate **5** was stirred for 24 hr in buffered acetic acid at 25°, an 89% yield of a mixture of acetates was obtained. Analysis by glpc (500-ft capillary column) showed the mixture was composed of 97% of an acetate **6**, 1.5% of minor acetate, and 1.5% of a mixture of several other compounds. Acetate **6** was purified by preparative glpc but the minor acetate was not studied due to its low yield. The main solvolysis product had the expected molecular formula, $C_{12}H_{14}O_2$ (mass spectrum), and its infrared spectrum was indicative of an acetate which contained a cis-disubstituted double bond. The nmr spectrum showed a two-proton quartet in the vinyl region; the coupling constant of 6.0 Hz established a cis-disubstituted double bond. Each band of this quartet was a finely split doublet (J = 2.2 Hz) indicating the presence of at least one allyl hydrogen on each side of the double bond. This conclusion was confirmed by a double resonance experiment in which irradiation of the band at 149 cps downfield from TMS, *i.e.*, the allylic hydrogen region, changed the vinyl resonance into a clean quartet with-

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(1) This investigation was supported in part by Grant No. GP-8700, National Science Foundation, and the Hoffmann-LaRoche Foundation.

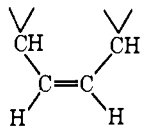
(2) National Institutes of Health Predoctoral Fellow, 1967–1970.

(3) R. M. Coates and J. L. Kirkpatrick, *J. Amer. Chem. Soc.*, **90**, 4162 (1968).

(4) S. Winstein, E. C. Friedrich, R. Baker, and Y. Lin, *Tetrahedron, Suppl.*, **8** [II], 621 (1966).

(5) M. A. Battiste, C. L. Deyrup, R. E. Pincock, and J. Haywood-Farmer, *J. Amer. Chem. Soc.*, **89**, 1954 (1967); H. Tanida, T. Tsuji, and T. Irie, *ibid.*, **89**, 1953 (1967).

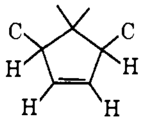
(6) W. G. Dauben, M. G. Buzzolini, C. H. Schallhorn, D. L. Whalen, and K. J. Palmer, *Tetrahedron Lett.*, 787 (1970).

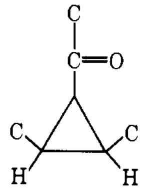

a

out the fine splitting. Thus, the presence of unit a is proved. The acetate function was cleaved by reduction with lithium aluminum hydride to yield an alcohol **7** which, in turn, was oxidized with chromium trioxide to ketone **8**. The ketone showed a 1700-cm^{-1} band in the infrared and a uv maximum at 196 nm (ϵ 8400). This latter absorption did not permit a decision to be rendered as to whether the carbonyl function was conjugated with a cyclopropane ring since the double bond by its strong end absorption could be masking the usual uv maximum of the conjugated cyclopropyl ketone. Therefore, the ketone **8** was treated with excess diimide, a reagent known to reduce *unhindered, nonpolar* double bonds,[7,8] and the dihydro ketone **9** was obtained. This product possessed a uv maximum at 202 nm (ϵ 5500), an absorption characteristic of a conjugated cyclopropyl ketone.[9] An infrared absorption band at 1702 cm^{-1} showed the carbonyl group was in an unstrained ring system. The value of the extinction coefficient in the ultraviolet as well as the absence of olefinic band in the Raman spectrum established the absence of any additional unsaturated linkages. Thus, being a saturated ketone, the empirical formula requires the presence of three rings in addition to the cyclopropane ring, *i.e.*, the solvolysis product must be a tetracyclic compound.

To obtain information with regard to the size of the ring containing the disubstituted double bond, the solvolysis alcohol **7** was converted to a mesylate ester which was reductively cleaved to yield the olefinic hydrocarbon **10**. Hydroboration of the double bond of **10** followed by chromium trioxide oxidation yielded two ketones in a 6:4 ratio. These ketones could not be completely separated by preparative glpc but each enriched ketone showed a single carbonyl absorption at 1745 cm^{-1} signifying that the original double bond was in a five-membered ring. In agreement with this conclusion as to ring size is the vicinal coupling constant of the vinyl protons of 6.0 Hz, a value characteristic of a cyclopentene.[10] The formation of two ketones indicated that this cyclopentene ring was not symmetrically substituted.

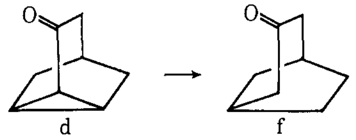
Attention was next turned to the cyclopropane ring. The substitution pattern could not be fully established by nmr spectroscopy but a two-proton multiplet centered at τ 8.9 indicated that each of the carbon atoms not attached to the carbonyl group carried a hydrogen atom; the presence of a geminal methylene group in the cyclopropane ring could be ruled out both by the chemical shifts and the spin-spin coupling pattern. Thus, at this point, the structural units b and c have been established for ketone **8**.

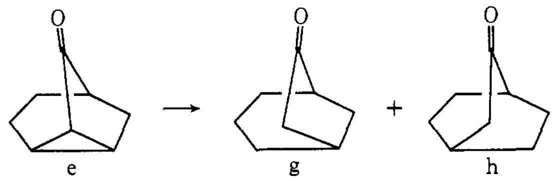

b


c

The presence of a cyclopropyl conjugated ketone in ketone **8** derived from the solvolysis alcohol **7** permitted further studies which established the complete structure of the ketone. Reduction of **8** with lithium in ammonia[11] gave two new ketones (relative ratio 7:3, not completely separated by glpc). The formation of two ketones showed that the starting material was not symmetrically substituted about the cyclopropyl carbonyl chromophore. The mixture of ketones had a single absorption in the infrared at 1710 cm^{-1}, corresponding to an unstrained ketone.

The nonpolar nature of the original olefinic linkage established that it was not in conjugation with the cyclopropyl carbonyl system and thus interpretation of the above reduction results needed only to consider the remaining eight carbon atoms in the molecule. These remaining carbon atoms must be arranged in a tricyclic structure; the carbonyl group must be conjugated with the cyclopropane ring so as to form a tetracyclic structure and yet must be in an unstrained ring. The entire structure must be so constructed that upon reductive cleavage of the cyclopropyl carbonyl system the two materials formed must be unstrained ketones. Starting with subunit c, only the tricyclic structures d and e can be written which will fit these data. The structure unit e can be ruled out on the basis of the infrared absorption requirements of the two possible ketones g and h which would have been formed upon reductive cleavage. The two ketonic ring systems g and h are known to display carbonyl absorptions at 1745 and 1735 cm^{-1}, respectively;[12] the products of the reaction both absorbed at 1710 cm^{-1}. The subunit d fits well the degradational results. The bicyclo[3.2.1]octan-3-one (f) ring system is known to display a carbonyl absorption at 1708 cm^{-1},[13] a value to be compared with 1710 cm^{-1} for the degradation ketones. Furthermore, the parent tricyclo-[2.2.2.0^{3,4}]octan-3-one is known[14] and it displays a


d f


e g h

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(7) E. J. Corey, W. L. Mocks, and D. J. Pasto, *Tetrahedron Lett.*, 347 (1961).

(8) W. C. Baird, B. Franzus, and J. Surridge, *J. Amer. Chem. Soc.*, **89**, 3449 (1967).

(9) W. G. Dauben and G. H. Berezin, *ibid.*, **89**, 3449 (1967).

(10) A. A. Bothner-By in "Advances in Magnetic Resonance," J. S. Waugh, Ed., Vol. 1, Academic Press, New York, N. Y., 1965, p 271.

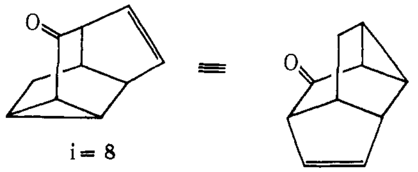
(11) (a) T. Norin, *Acta Chem. Scand.*, **19**, 1289 (1965); (b) W. G. Dauben and E. J. Deving, *J. Org. Chem.*, **31**, 3794 (1966).

(12) H. O. House, S. G. Boots, and V. K. Jones, *ibid.*, **30**, 2519 (1965).

(13) W. R. Moore, W. R. Moser, and J. E. LaPrade, *ibid.*, **28**, 2200 (1963).

(14) W. von E. Doering, E. T. Fossel, and R. L. Kaye, *Tetrahedron*, **21**, 25 (1965).

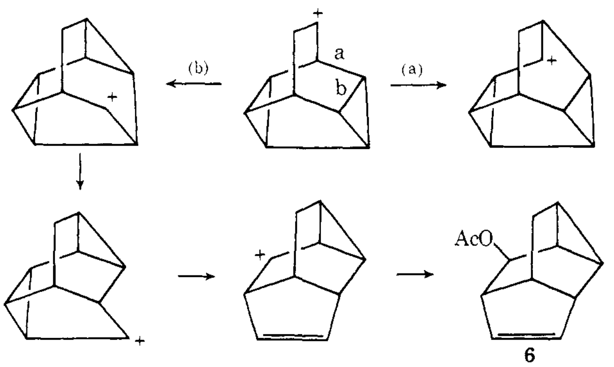
carbonyl absorption at 1703 cm^{-1} and a uv max at 199 nm (ϵ 7700),[15] values to be compared with 1702 cm^{-1} and 202 nm (ϵ 5500) for the dihydro ketone **9**. Thus, the subunit d represents the tricyclic system to which the cyclopentene ring must be attached. This attachment can only be on methylene carbons of this subunit since after the attachment of the ring there must be allylic hydrogen atoms in the structure. The additional requirement that the structure be unsymmetrical leads to the unique structure i for the ketone **8**.



i= 8

i

With the structure of the rearrangement product established, the mechanism of the process can be considered. The solvolytic process could begin with a 1,2 migration (bond a) or with a homocyclopropylcarbinyl rearrangement (bond b). The structure of the product clearly establishes the fact that the latter process is, indeed, favored. The logical subsequent rearrangements leading to the product are shown below.



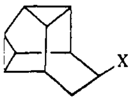
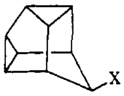
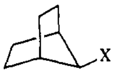
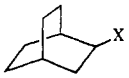
6

Rate Study of the Solvolysis of Tosylate 6. The tosylate **6** reacted with buffered acetic acid with first-order rate constants of $k = 7.02 \times 10^{-5}$ sec^{-1} at 25.0° (two runs) and $k = 5.0 \times 10^{-4}$ sec^{-1} at 45.0° (one run). The reaction was followed by titration of unreacted sodium acetate buffer. The infinity titer at 25.0° was within 3% of the theoretical value after 10 half-lives indicating little internal return of tosylate anion to a species of a much less reactivity.

Table I indicates how well the new pentacyclic system and other related compounds fit the Foote correlation.[16]

If the carbonyl stretching frequency would be an absolute measure of the difficulty of the carbinyl position to achieve sp^2 hybridization, one would predict ring system **1** to solvolyze about as fast as the 7-norbornyl derivative. This great discrepancy has been attributed to homocyclopropyl participation in the transition state.[6] The infrared spectrum of the ketone in this present pentacyclodecane system correlates with a rate of 0.2 times cyclohexyl tosylate for tosylate **5**. The experimentally determined rate is over 10^3 faster suggesting that participation of an alkyl bond in the transition state is significantly accelerating the rate. The rate is also about 20 times faster than the parent bicyclo[2.2.2]octyl system, another compound which departs considerably from its expected solvolysis rate. It thus appears that homocyclopropyl participation is enhancing the solvolysis rate of tosylate **5** from the value to be expected from just strain release. The magnitude of the enhancement is considerably less than that found for the systems which yield a cation which can rapidly rearrange to identical structures, the limiting case being a degenerate cation.

Table I. Relative Solvolysis Rates of Cyclic Compounds

Compound	Carbonyl stretching frequency of corresponding ketone, cm^{-1}	Predicted rate (25°) relative to cyclohexyl tosylate	Actual rate (25°) relative to cyclohexyl tosylate
 X	1725	0.2	1.4×10^3
 X	1770	5×10^{-7}	10^{6} [a]
 X	1773	10^{-7}	10^{-7} [b,c]
 X	1731	3×10^{-2}	70[c]

[a] See ref 3. [b] See ref 4. [c] See ref 11.

Experimental Section

All tlc work was done with silica gel PF$_{254}$ (E. Merck A.G.) on microscopic slides. Vapor phase chromatography was carried out with a Wilkins Aerograph, Model A-90-P, with a 10% SE-30 column. Analyses were made using a 500 ft, 0.03 in. capillary column coated with OV-101 in a custom-made Teranishi apparatus.

Combustion analyses were performed by the Microanalytical Laboratory, College of Chemistry, University of California, Berkeley.

Pentacyclo[4.4.0.0^{2,4}.0^{3,8}.0^{5,7}]decane 9-Tosylate (5). A solution of pentacyclo[4.4.0.0^{2,4},0^{3,8}.0^{5,7}]decan-9-ol (**4**)[6] (500 mg, 3.4 mmol) in 4 ml of dry pyridine was cooled in an ice bath. Then tosyl chloride (1.3 g, 6.8 mmol) was added to the stirred solution. The reaction was stirred until homogeneous and then placed overnight in a refrigerator. The reaction mixture was diluted with cold water and extracted with ether. The combined ether extracts were washed with cold 10% sulfuric acid, water, and saturated sodium bicarbonate solution, and then dried. The solvent was removed by evaporation to give 0.99 g (95%) of the crude tosylate **5**, a slightly brown, viscous oil. The tosylate could be recrystallized in pentane–ether to give white crystals: mp 65.0–66.5°; ir (CCl$_4$) 1175, 1158 cm^{-1} (sulfonate ester); nmr (CCl$_4$) τ 2.50 (4 H, g), 5.25 (1 H, m), a singlet on top of a multiplet at 7.57, together accounting for 5 H, 8.40 (8 H, m); mass spectrum *m/e* 302.

Anal. Calcd for $C_{17}H_{18}O_3S$: C, 67.52; H, 5.95. Found: C, 67.20; H, 6.05.

Sodium–Liquid Ammonia Reduction of Pentacyclo[4.4.0.0^{2,4}.0^{3,8}.0^{5,7}]decane 9-Tosylate (5). A solution of 0.40 g of sodium in about 40 ml of liquid ammonia was prepared in a three-necked flask equipped with a Dry Ice condenser, fitted with a potassium hydroxide drying tube, and containing a magnetic stir bar. A solution of 200 mg (0.66 mmol) of tosylate **5** in 5 ml of ether was added, and the reaction mixture was allowed to stir for 2 hr. Solid ammonium chloride was added to decompose the excess sodium, and then 10 ml of water was carefully added. The reaction mixture was allowed to stir for 1 additional hr, and then was diluted with

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(15) W. G. Dauben and J. Frei, unpublished observation.

(16) C. S. Foote, *J. Amer. Chem. Soc.*, **86**, 1853 (1964).

ether. The ether solution was washed with 5% hydrochloric acid solution and water, and then dried. The solvent was removed by evaporation to yield 90 mg of a white solid. The nmr, ir, and mass spectra of the product were identical with those of the alcohol **4**.

Preparative Acetolysis of Pentacyclo[4.4.0.0^{2,4}.0^{3,8}.0^{5,7}]decane 9-Tosylate (5). The tosylate **5** (500 mg, 1.65 mmol) and 160 mg (2.0 mmol) of sodium acetate were dissolved in 15 ml of anhydrous acetic acid. The resulting solution was stirred at 25° for 25 hr (approximately ten half-lives). The reaction mixture was diluted with water and extracted with pentane. The combined pentane extracts were washed with water and sodium bicarbonate solution, and then dried. The solvent was removed by evaporation to yield 280 mg (89%) of a clear oil. This oil was examined by glpc (500-ft column, OV-101 substrate, 120°) and found to contain 97% of one acetate **6**. This acetate was purified by preparative glpc to yield a clear oil: ir (CCl_4) 1730, 1245, 710 cm^{-1}; nmr (CCl_4) τ 4.20 (2 H, AB quartet, allylic coupling J_{AB} = 6.0 Hz, $J_{allylic}$ = 2.2 Hz), 5.44 (1 H, d, J = 5.0 Hz), 8.05 (3 H, s), 8.9 (2 H, m); mass spectrum, *m/e* 190, 148, 130.

Determination of the Acetolysis Rate of Pentacyclo[4.4.0.0^{2,4}.0^{3,8}.0^{5,7}]decane 9-Tosylate (5). Anhydrous acetic acid was prepared by mixing reagent grade acetic acid with 3% acetic anhydride, stirring overnight, and then distilling. To the center cut, bp 117–118°, was added 1% acetic anhydride. Reagent grade sodium acetate was dried at 170° for 5 days before use.

(a) The rate determinations at 25.0° were conducted according to Winstein, *et al.*[17] A solution of anhydrous acetic acid containing a known excess of sodium acetate was placed in a 25.0° temperature bath for 1 hr. Then a carefully weighed amount of pure tosylate **5** was added to the solution; the reaction vessel was shaken vigorously and then placed back in the temperature bath. The point of addition of the tosylate was taken as time = 0. Periodically aliquots were taken from the reaction solution and diluted with cold ether, and the remaining sodium acetate buffer was titrated with a standard perchloric acid solution in acetic acid. Two runs were made, and the rate constant obtained was $k = 7.02 \times 10^{-5}$ sec^{-1}. The calculations were made with the aid of a computer using a least-squares program. Good first-order plots were obtained in both cases, and the infinity titer (after ten half-lives) was within 3% of the theoretical value both times.

(b) The rate determination at 45.0° was made according to Wiberg, *et al.*[18] Known amounts of standard sodium acetate were added to five test tubes. Each tube was marked at a volume of 4.0 ml, and the solvent was made to this volume with anhydrous acetic acid. Two drops of a 1% solution of bromophenol blue in acetic acid were added to each tube, and the tubes were stoppered. The tubes were placed in a 45.0° temperature bath for at least 20 min. A 100-μl portion of a 0.622 *M* solution of tosylate **5** in CCl_4 was injected into each tube. The stoppered tubes were shaken vigorously for a few seconds and immediately replaced in the temperature bath. The time of the color change for each tube was noted. The first-order rate constant obtained in this manner was $k = 5.0 \times 10^{-4}$ sec^{-1}.

Reduction of the Solvolysis Acetate (6). Formation of the Solvolysis Alcohol (7). The crude acetate **7** made from 500 mg (1.65 mmol) of tosylate was reduced with excess lithium aluminum hydride in ether in the usual manner. The product was 144 mg (59% from tosylate) of the white solvolysis alcohol **7**: mp 124–126° after recrystallization in hexane; ir (CCl_4) 3650–3350 cm^{-1}; nmr (CCl_4) τ 4.20 (2 H, AB quartet, allylic coupling, J_{AB} = 6.0 Hz, $J_{allylic}$ = 2.0 Hz), 6.32 (1 H, d, J = 4.5 Hz); mass spectrum, *m/e* 148, 130.

Anal. Calcd for $C_{10}H_{12}O$: C, 81.04; H, 8.16. Found: C, 81.27; H, 8.17.

Oxidation of the Solvolysis Alcohol (7). Formation of the Solvolysis Ketone (8). A solution of 25 mg (0.16 mmol) of the solvolysis alcohol **7** in 2 ml of acetone was oxidized at 0° according to the usual Jones oxidation procedure.[19] This procedure yielded 20 mg of a clear oil. A sample was purified by preparative glpc to give the pure solvolysis ketone **8**: ir (CCl_4) 3035, 2920, 1700 cm^{-1}; mass spectrum, *m/e* 146, 118; uv max (95% ethanol) 196 nm (ϵ 8400).

Reduction of the Solvolysis Ketone 8 with Diimide.[7,8] To a stirred suspension of 754 mg (3.88 mmol) of dipotassium azodicarboxylate[20] in 10 ml of methanol containing 150 mg (1.05 mmol) of solvolysis ketone **8** was added dropwise 472 mg (7.88 mmol) of acetic acid in 5 ml of methanol. The reaction was stirred for 30 min at room temperature. The reaction mixture was diluted with water and extracted with pentane. The pentane extracts were dried and the solvent removed by evaporation to yield 73 mg (47%) of an oily solid. The oily solid was essentially pure according to glpc. A sample was purified by preparative glpc to give the pure reduced ketone **9**: ir (CCl_4) 3050, 2960, 1702 cm^{-1}; mass spectrum, *m/e* 148, 130; uv max (95% ethanol) 202 nm (ϵ 5500).

Lithium–Ammonia Reduction of the Solvolysis Ketone (8). Following the standard procedure[11] using 5 ml of dry ammonia and 35 mg of lithium, a solution of 60 mg of solvolysis ketone **8** in 2 ml of dry ether was reduced for 1 hr and processed in the usual fashion. The clear oily product (60 mg) showed a single ir band at 1710 cm^{-1}; glpc analysis showed two products in a ratio of 7:3. The main peak was collected by glpc and had a purity greater than 95%: ir (CCl_4) 2940, 1710, 1605 cm^{-1}; mass spectrum, *m/e* 148, 120 (M − CO), 91 ($C_7H_7^+$), 79, 78.

Removal of the Hydroxyl Function of the Solvolysis Alcohol (7) (Formation of Hydrocarbon 10). A solution of the solvolysis alcohol **7** (200 mg, 13.5 mmol) in 5 ml of benzene was cooled in an ice bath. Mesyl chloride (162 mg, 1.42 mmol) was added to the stirred, cooled solution followed by triethylamine (150 mg, 1.49 mmol). The ice bath was removed and the solution stirred for 10 more min. The mixture was filtered and the triethylammonium chloride was washed with more benzene. The solvent was removed from the combined reaction solution and washings by evaporation to yield 232 mg (77%) of a slightly yellow oil, ir (CCl_4) 1360, 1340, 1175 cm^{-1} (sulfonate ester).

The crude mesylate was dissolved in 20 ml of dry ether and reduced in ether solution with lithium aluminum hydride in the usual manner. All of the ether was not removed from the product by rotary evaporator because of the expected volatility of the resulting hydrocarbon. Examination by tlc showed mostly one spot with a R_f about 2.5 times that of the starting alcohol. The crude product was purified by glpc to yield a hydrocarbon **10** (estimated yield 75% from mesylate according to glpc): ir (CCl_4) 3040, 2940, 2860, 1605, 729 cm^{-1}; nmr (CCl_4) τ 4.20 (2 H, AB quartet allylic coupling, J_{AB} = 6.0 Hz, $J_{allylic}$ = 1.9 Hz), 6.5 (3 H, m), 9.15 (2 H, m); mass spectrum, *m/e* 132.

Hydroboration–Oxidation of the Solvolysis Hydrocarbon (10). A solution of 25 mg (0.2 mmol) of crude solvolysis hydrocarbon **10** in 1 ml of anhydrous tetrahydrofuran was cooled in an ice bath. Then 0.6 ml of a 1.0 *M* solution of diborane in tetrahydrofuran was added to the stirred solution with a syringe under a nitrogen atmosphere. The solution was stirred for 20 min at 0.5° and for 18 hr at room temperature. The excess diborane was destroyed by the slow addition of water. Then 0.1 ml of 3 *N* sodium hydroxide was added followed by 0.1 ml of 30% hydrogen peroxide. The mixture was stirred for 4 hr and then diluted with ether. The aqueous solution was drawn off and the organic phase washed with water, a 5% ferrous sulfate solution containing a trace of sulfuric acid, and again with water. The ether solution was dried, and the solvent was removed by evaporation. The residue was oxidized in acetone solution at 0° with Jones reagent.[19] This gave 13 mg (50%) of a clear oil which consisted of two components on glpc. The two products were in a ratio of about 6:4 and could not be completely separated on glpc. The infrared spectrum of a glpc-purified mixture of the two components showed a strong, broad band at 1745 cm^{-1}.

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