dried. Removal of solvent gave 60.9 g (100%) of **2** which solidified on standing, mp 29.5–30°. Glc (silicone SE-30) showed one major peak (98.4%, **2**) and one minor peak (1.6%, **1**): ir of **2** (neat) 3.4, 3.5, 6.83, 7.2, 7.29, 7.39, 8.0, 8.29, 8.42, 8.6, 8.99, 9.1, 9.25, 9.35, 9.55, 10.55, 10.9, 12.1, 12.7, 14.1 μ; nmr δ 1.32 and 1.5 [two s, 28, $-(\mathrm{CH_2})_{14}-$], 3.89 (s, 4, $-\mathrm{OCH_2CH_2O}-$); mass spectrum *m/e* 268 $(\mathrm{M})^+$, 99, 225, 155, 55, 84, 41.

Anal. Calcd for $\mathrm{C_{17}H_{32}O_2}$: *m/e* 268.2402. Found: *m/e* 268.2403.

Ethylene Ketal of 2-Bromocyclopentadecanone (3).—To a cold (0°) stirred solution of **2** (60.9 g, 0.228 mol, from the above experiment) in anhydrous tetrahydrofuran (1.5 l.) was added rapidly phenyl trimethylammonium tribromide (85.7 g, 0.228 mol). An orange color was developed which gradually disappeared after 1 hr. The stirring was continued for an additional 1 hr at 0°. The mass was poured into saturated sodium bicarbonate solution (600 ml) and was stirred for 30 min. Most of the tetrahydrofuran was removed under reduced pressure without heating. The aqueous layer was extracted with ether. The ethereal solution was washed with saturated sodium chloride solution and dried. Removal of solvent gave 83 g (107%) of crude bromide **3**. Glc (silicone SE-30) showed a major peak and a minor peak due to **1** and **2**, respectively. Bromide **3** did not elute from the glc column: ir of **3** (crude) 5.89 (C=O of regenerated **1**) and 9.5 μ (–COC– of ketal **3**); nmr δ 1.1–2.1 (broad m with s at 1.32 and a shoulder at 1.3), 2.66 (small t), 3.42 (small t), 3.68 (small t), 3.8–4.36 (m, –CHBr and $-\mathrm{OCH_2CH_2O}-$), 4.5–4.7 (small m); mass spectrum *m/e* 346 $(\mathrm{M})^+$ and 348 $(\mathrm{M}+2)^+$.

Anal. Calcd for $\mathrm{C_{17}H_{31}O_2Br}$: *m/e* 346.1507. Found: *m/e* 346.1508.

Ethylene Ketal of 2-Cyclopentadecen-1-one (4.)—A 2.5-l. flask equipped with stirrer, reflux condenser, thermometer, and a nitrogen inlet tube was charged with crude bromide **3** (83 g, 0.2 mol, from the previous experiment) and 1,5-diazabicyclo[4.3.0]non-5-ene (89.28 g, 0.72 mol). The mixture was heated at 110° for 64 hr, cooled, and poured into water. The aqueous mixture was extracted with ether. The combined ether extracts were washed successively with water and saturated sodium chloride solution and dried. Removal of solvent gave 56.5 g of crude oil which was chromatographed on deactivated silicic acid (600 g); 10–20% ether in hexane (1.5 l. per fraction) eluted 47.1 g (74.6%) of **4**. Glc (Carbowax 20M) analysis showed one major peak (88%, **4**) and two minor peaks due to **1** (5%) and **2** (7%): ir of **4** (neat) 3.45, 3.5, 6.0, 6.85, 6.9, 7.2, 7.3, 7.4, 7.65, 7.79, 7.99, 8.49, 8.9, 9.5, 10.25, 10.55, 12.4, 13.4, and 13.85 μ; nmr δ 1.1–2.3 (major s at 1.3, and broad m at 1.7 and 2.1, 24, $-\mathrm{CH_2}-$), 3.9 (s, 4, $-\mathrm{OCH_2CH_2O}-$), 5.1–6.0 (d at 5.3, 1 and m at 5.7, 1, –CH=CHCOO); mass spectrum *m/e* 266 $(\mathrm{M})^+$, 125, 99, 55, 41.

Anal. Calcd for $\mathrm{C_{17}H_{30}O_2}$: *m/e* 266.2247. Found: *m/e* 266.251.

2-Cyclopentadecen-1-one (5).—A solution of **4** (46.1 g, 0.17 mol, from the previous experiment) and *p*-toluenesulfonic acid monohydrate (6.65 g, 0.035 mol) in water (100 ml) and acetone (50 ml) was stirred for 16 hr at room temperature. Most of the acetone was removed under reduced pressure, and the residue was poured into water. The aqueous mixture was extracted with ether. The ether extracts were washed successively with water and saturated sodium chloride solution and dried. Removal of solvent under reduced pressure yielded 38.5 g (100%) of crude **5**. Glc analysis (silicone SE-30) showed one major peak (74%, **5**) and one minor peak (26%, **1**): ir of **5** (neat) 3.4, 3.5, 5.82, 5.9, 5.99, 6.15, 6.77, 6.82, 6.9, 7.2, 7.4, 7.8, 8.25, 8.75, 8.85, 9.25, 9.55, 10.2 μ; nmr δ 1.1–2.0 (m, 20 with a s at 1.3, $-\mathrm{CH_2}-$), 2.1–2.54 (m, 4, $-\mathrm{CH_2COC{=}CCH_2}$), 6.08–7.0 (d, at 6.16, 1 and m at 6.8, 1, –CH=CHCO); mass spectrum *m/e* 222 $(\mathrm{M})^+$ 41, 55, 81, 67, 68.

Anal. Calcd for $\mathrm{C_{15}H_{26}O}$: *m/e* 222.1983. Found: *m/e* 222.1986.

***dl*-Muscone (6).**—A solution of **5** (37.5 g, 0.169 mol, from the previous experiment) in anhydrous ether was added slowly over a period of 1 hr to a stirred mixture of methylmagnesium bromide (62 ml, 0.185 mol) and cuprous chloride (11.16 g) in anhydrous ether (750 ml) at 10°. After the addition was completed, the reaction mixture assumed a dark grayish-green color and stirring was continued for 2 hr at 10°. Cold aqueous 10% hydrochloric acid (200 ml) was added slowly, and the organic phase was separated. The aqueous layer was extracted with ether. The combined ethereal solutions were washed successively with saturated sodium bicarbonate solution, water, and saturated sodium chloride solution (50 ml) and dried. Removal of solvent under reduced pressure gave 40.2 g of crude oil which was chromatographed on deactivated silicic acid (600 g): 5% and 7.5% ether in hexane (1 l. per fraction) eluted 32.2 g (81.1%; an overall 61.5% from exaltone **2**) of muscone (**6**). Glc analysis (Carbowax 20M and silicone SE-30) showed one peak. This material was further distilled to obtain 27 g of **6**: bp 100–101° (0.09 mm); ir of **6** (neat) 3.41, 3.5, 5.84, 6.82, 7.09, 7.29, 7.8, 8.35, 8.85, 9.2, 9.45, 9.8, and 14.0 μ; nmr δ 0.92 (d, $J = 6$ Hz, 3, $\mathrm{CH_3CH}-$), 1.1–2.0 (m, 23, with s at 1.3), 2.1–2.5 (m, 4, $-\mathrm{CH_2COCH_2}$); mass spectrum *m/e* 238 $(\mathrm{M})^+$, 41, 55, 85, 69, 71, 43.

(All these spectral data were superimposable with those of natural muscone and also with synthetic *dl*-muscone made from 1,9-cyclohexadecadiene.[2])

Anal. Calcd for $\mathrm{C_{16}H_{30}O}$: *m/e* 238.2296. Found: *m/e* 238.2298.

Reaction of 3 with Potassium *tert*-Butoxide.—A mixture of **3** (1.15 g, 0.0033 mol), potassium *tert*-butoxide (1.11 g, 0.01 mol), and anhydrous *tert*-butyl alcohol (20 ml) was refluxed for 20 hr. Most of the solvent was removed under reduced pressure, water (20 ml) was added, and the mixture was extracted with ether. The combined ether extracts were washed with saturated sodium chloride solution and dried. Evaporation of solvent under reduced pressure gave 0.8 g of yellow oil. Glc (Carbowax 20M) gave one major peak due to **7**: ir (neat) 3.42, 3.5, 5.92, 6.9, 7.4, 7.82, 8.09, 8.3, 8.6, 8.7, 9.02, 9.32, 9.85, 10.8, and 11.15 μ; nmr δ 1.2–1.8 (m, 22, with s at 1.35, $-\mathrm{CH_2}$), 2.04 (m, 4, $-\mathrm{CH_2C{=}CCH_2}$), 3.98 (s, 4, $-\mathrm{OCH_2CH_2O}-$); mass spectrum *m/e* 266 $(\mathrm{M})^+$, 99 (base peak).

A solution of **3** (1.15 g) and potassium *tert*-butoxide (1.11 g) in dimethyl sulfoxide (20 ml) was refluxed for 10 hr. After usual work-up, a crude oil (1 g) was obtained. Glc showed a major peak due to **7**.

Anal. Calcd for $\mathrm{C_{17}H_{30}O_2}$: *m/e* 266.2295. Found: *m/e* 266.2248.

Registry No.—**1**, 502-72-7; **2**, 184-41-8; **3**, 32247-06-6; **4**, 32247-07-7; **5**, 32247-08-8; **6**, 956-82-1; **7**, 32304-18-0.

Acknowledgment.—The authors express their gratitude to Dr. W. I. Taylor for his constant encouragement during this work. We also gratefully acknowledge Professor H. H. Wasserman for providing us with the detailed procedure of dehydrobromination with diazabicyclononene. We also thank Mr. H. Bondarovich for high resolution mass spectral analyses and Mr. M. Jacobs for nmr analyses.

Transannular Ring Closure by Reduction of Cyclooctane-1,5-diones. Synthesis of a Bisnoradamantan-1-ol

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A reasonable approach to the synthesis of the series of theoretically interesting bridgehead olefins **3**[1] appeared to be double bond formation from the glycols **2**, by one of a number of known reactions.[2-4] More-

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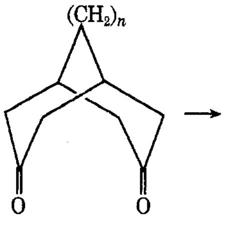
(1) For a recent example of a polycyclic molecule with a similar "strained" double bond, see N. M. Weinshenker and F. D. Greene, *J. Amer. Chem. Soc.*, **90**, 506 (1968).

(2) E. J. Corey, F. A. Carey, and R. A. E. Winter, *ibid.*, **87**, 934 (1965).

(3) J. N. Hines, M. J. Peagram, G. H. Whitham, and M. Wright, *Chem. Commun.*, 1593 (1968).

(4) F. W. Eastwood, K. J. Harrington, J. S. Josan, and J. L. Pura, *Tetrahedron Lett.*, 5223 (1970).

over, the facility of 1,5-transannular reactions in cyclooctane rings[5] and instances of intramolecular pinacol formation on reduction of diketones[6] suggested that the required diols (**2**) might be synthesized from the bridged cyclooctane-1,5-diones (**1**).



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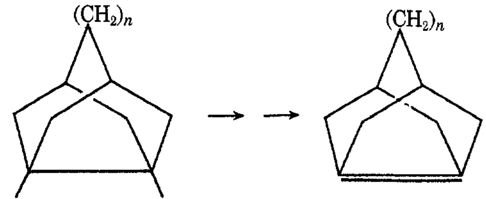


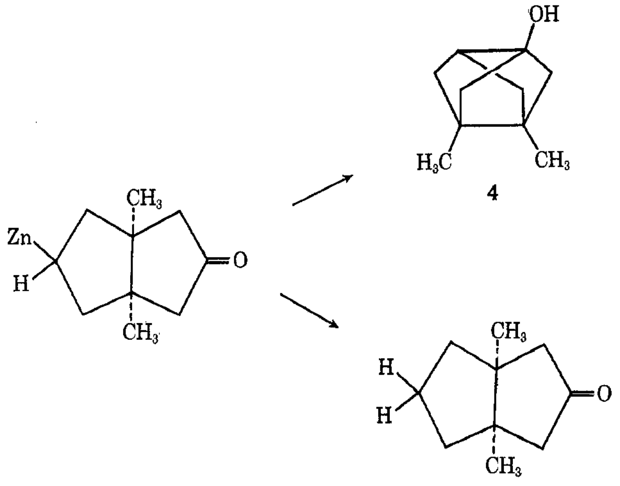
2 **3**

As a model system we first examined the reduction of cyclooctane-1,5-dione[7] itself. Reaction of the diketone with Zn–HCl in acetic anhydride[8] led to the isolation of a crystalline diacetate. Treatment with sodium methoxide in methanol liberated the bicyclic diol,[9] homogeneous by glc, in 60% overall yield after recrystallization from hexane, mp 61.5–62.0°. Alternatively the diol could be obtained directly and in essentially quantitative yield by reduction of the diketone with zinc amalgam in aqueous hydrochloric acid.[11]

Similarly, in the bridged system, reduction of bicyclo[3.3.1]nonane-3,7-dione (**1**, $n = 1$)[12] gave tricyclo[3.3.1.0^{3,7}]nonane-3,7-diol (**2**, $n = 1$), mp 301–303°.[13]

In contrast, in the $n = 0$ series the readily available 1,5-dimethylbicyclo[3.3.0]octane-3,7-dione[14] on reduction in acetic anhydride did not give the corresponding tricyclic diol. Instead, after deacetylation and purification by chromatography followed by sublimation,[15] a mono alcohol (m/e 152, correct analysis for $C_{10}H_{16}O$) was obtained in 50% yield, mp 108–109°. Its nmr spectrum ($CDCl_3$) showed δ 1.10 (s, 6 H), 1.2–1.9 (m, 8 H), 2.10 (t, $J = 3$ cps, 1 H) and one exchangeable proton, consistent with its formulation as 3,7-dimethyltricyclo[3.3.0.0^{3,7}]octan-1-ol (**4**). However, when the reduction was carried out in aqueous solution, **4** was only a minor product (10% yield); the major one was 1,5-dimethylbicyclo[3.3.0]octane, the hydrocarbon formed by "normal" Clemmensen reduction of the diketone.[16]

A mechanism for the formation of **4**, which also rationalizes the dependence of its yield on the solvent, is shown below. In the absence of appreciable interaction between the carbonyl groups in the diketone, due to the $n = 0$ bridge, Clemmensen reduction is expected to occur. A postulated intermediate in the reduction is the organozinc compound shown below.[6] It could undergo intramolecular addition to the remaining carbonyl to give **4** or it could be protonated to give **5**. Which course the reaction takes would depend on the availability of protons from the solvent. Clearly the path leading to **5** would be favored in aqueous solution; and **5** is found, as expected, to undergo reduction to the observed bicyclic hydrocarbon in the aqueous reaction.



4

5

Not only is **4** of interest on account of the unusual mechanism by which it is formed, it is the first molecule with a bisnoradamantane skeleton in which the bridgehead carbonium ion can be studied.[17] The geometry of this ion should be particularly unfavorable; in fact, Bingham and Schleyer have calculated that acetolysis of even the triflate to produce the bridgehead carbonium ion should have a rate constant on the order of 10^{-11} sec^{-1} at 200°.[18] Our preliminary results on the solvolysis of the tosylate indicate that the reaction appears to be accelerated by fission of the bond between C-2 and C-3, which both circumvents the bridgehead ion and relieves strain present in the bisnoradamantane skeleton.[19] Another manifestation of the strain present in this system is the transformation of **4** to **5** by potassium *tert*-butoxide in refluxing *tert*-butyl alcohol.[20]

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[5] Review: A. C. Cope, M. M. Martin, and M. A. McKervey, *Quart. Rev., Chem. Soc.*, **20**, 119 (1966).

[6] Review: J. G. St. C. Buchanan and P. D. Woodgate, *ibid.*, **23**, 522 (1969).

[7] G. I. Glover, R. B. Smith, and H. Rapoport, *J. Amer. Chem. Soc.*, **87**, 2003 (1965).

[8] T. J. Curphey, C. W. Amelotti, T. P. Layloff, R. L. McCartney, and J. H. Williams, *ibid.*, **91**, 2817 (1969).

[9] The expected cis ring fusion was documented by the formation of the cyclic acetal of benzaldehyde, which on reaction with *n*-butyllithium[3] in ether gave $\Delta^{1,5}$-bicyclo[3.3.0]octene.[10]

[10] (a) L. A. Paquette and R. W. Hauser, *J. Amer. Chem. Soc.*, **91**, 3870 (1969); (b) E. J. Corey and E. Block, *J. Org. Chem.*, **34**, 1233 (1969); (c) E. Block, Ph.D. Thesis, Harvard University, 1967.

[11] E. Wenkert and J. E. Yoder, *J. Org. Chem.*, **35**, 2986 (1970).

[12] A. R. Gagneux and R. Meier, *Tetrahedron Lett.*, 1365 (1969).

[13] While this work was in progress, the preparation of this diol (reported mp 297–298°) by transannular ring closure on photoreduction of the diketone was reported by T. Mori, K. H. Kimoto, and H. Nozaki, *ibid.*, 2419 (1970).

[14] U. Weiss and J. M. Edwards, *ibid.*, 4885 (1968).

[15] From the crude product 1,5-dimethylbicyclo[3.3.0]octan-3-one (**5**) was also isolated in 10% yield.

[16] The hydrocarbon is not produced from **4**, which is stable under the reaction conditions.

[17] Solvolysis of 2-chloro- and a derivative of 2-hydroxybisnoradamantane has been studied: (a) P. K. Freeman, R. B. Kinnel, and J. D. Ziebarth, *Tetrahedron Lett.*, 1059 (1970); (b) R. R. Sauers and B. R. Sickles, *ibid.*, 1067 (1970).

[18] R. C. Bingham and P. v. R. Schleyer, *J. Amer. Chem. Soc.*, **93**, 3189 (1971).

[19] W. T. Borden and C. Schmidt, unpublished results. We are also studying the solvolysis of a derivative of the unsubstituted bisnoradamantan-1-ol.

[20] Similar facile reketonizations are observed in other strained systems: (a) cyclopropanols, C. H. DePuy, *Accounts Chem. Res.*, **1**, 33 (1968); (b) bird cage alcohols, R. Howe and S. Winstein, *J. Amer. Chem. Soc.*, **87**, 915 (1965); T. Fukunaga, *ibid.*, **87**, 917 (1965); (c) strained cyclobutanols, K. B. Wiberg, J. E. Hiatt, and K. Hsieh, *ibid.*, **92**, 544 (1970).

A study of the stereochemistry of this reaction has been reported separately.[21]

Experimental Section

All nmr spectra were run on a Varian A-60 instrument using $CDCl_3$ as solvent. Melting points were taken in sealed capillaries and are uncorrected. All reactions were carried out under an atmosphere of nitrogen.

General Procedure for Zn-HCl Reductions.—Acetic anhydride (40 ml) was saturated with HCl at -5 to $-10°$ and 1 g of diketone was added; 10 g of activated zinc dust[22] was added slowly in portions over 3 hr with vigorous mechanical stirring so that the temperature of the reaction mixture remained below $-5°$. If the temperature was allowed to rise higher than this, the reaction became uncontrollable and the temperature rose rapidly to 40–50°. On completion of the addition the mixture was stirred for 2 hr longer at $-5°$ and then filtered quickly through a prechilled funnel to remove the zinc residue, which was washed with cold acetic anhydride.

The diacetate was isolated by diluting the filtrate with 40 ml of water and adding solid Na_2CO_3 until the pH was neutral. The solution was extracted with three 100-ml portions of CH_2Cl_2, and the combined organic layers were washed with two 100-ml portions of water and dried over $MgSO_4$. Evaporation of solvents under reduced pressure gave the crude product.

This was converted to the free alcohol by stirring in 15 ml of methanol, 2 *N* in $NaOCH_3$, for 1 hr at room temperature. The solution was neutralized with excess Amberlite IR 120 (pyridinium form[23]), and the solution was decanted from the resin, which was washed with two 20-ml portions of methanol. Removal of the methanol under reduced pressure gave the crude alcohol, which was purified by chromatography, recrystallization, or sublimation.

Alternatively the alcohol could be isolated directly by adding the acetic anhydride filtrate to 200 ml of cold methanol and leaving the solution overnight. The solvents were removed under reduced pressure, and the oily residue was taken up in CH_2Cl_2 and washed with two 50-ml portions of H_2O, two 50-ml portions of 10% $NaHCO_3$, and 50 ml of H_2O. The organic layer was dried over $MgSO_4$ and evaporated under reduced pressure to give the crude alcohol.

Reactions using zinc amalgam in aqueous solution were carried out using the procedure of Wenkert and Yoder.[11]

Bicyclo[3.3.0]octane-1,5-diol.—The diol showed a symmetrical multiplet centered at δ 1.8 (12 H) and two exchangeable protons. An analytical sample, mp 61.5–62.0°, was obtained by recrystallization from hexane, mass spectrum *m/e* 142.

Anal. Calcd for $C_8H_{14}O_2$: C, 67.57; H, 9.92. Found: C, 67.61; H, 9.78.

$\Delta^{1,5}$-Bicyclo[3.3.0]octene.[10]—The benzaldehyde acetal of bicyclo[3.3.0]octane-1,5-diol was prepared by refluxing a solution of 426 mg (3 mmol) of the diol and 318 mg (3 mmol) of benzaldehyde in 15 ml of benzene with a catalytic amount (20 mg) of *p*-toluenesulfonic acid for 3 hr. The cooled solution was stirred with solid K_2CO_3 and the solvents were evaporated. The residue was chromatographed over 10 g of neutral alumina to remove trace amounts of unreacted starting material. Elution with hexane gave 620 mg (90% yield) of a straw-colored liquid, nmr δ 1–2.5 (m, 12 H), 5.80 (s, 1 H), and 7.4 (m, 5 H).

To a solution of 230 mg (1 mmol) of the benzaldehyde acetal in 20 ml of ether at 0° was added 1.6 ml of 1.3 *M* *n*-butyllithium in pentane (2.1 mmol). The solution was stirred for 24 hr at 0° and then 2 ml of H_2O was added. The organic layer was separated, the water was washed with two 2-ml portions of ether, and the combined ether washings were washed with 2 ml of H_2O and 2 ml of brine. After drying over $MgSO_4$, most of the solvent was removed by distillation through a 25-cm Vigreux column. Then 1 ml of CH_2Cl_2 was added, the stillhead was attached directly to the pot, and the distillation was continued until no more solvent was collected at a pot temperature of 80°. The pot was allowed to cool to room temperature and the distillation was continued under vacuum (water aspirator $\approx$15 mm) while cooling the receiver in liquid nitrogen. The pot was briefly heated to 60° before the distillation was discontinued; 170 mg of volatile material was collected and analyzed by nmr, which showed three singlets, one for CH_2Cl_2, one for cyclohexane (present in the commercial *n*-butyllithium solution), and one at δ 2.18 from the $\Delta^{1,5}$-bicyclo[3.3.0]octenes.[10b] A pure sample of this material was isolated by glc and had an ir spectrum identical with that reported by Block.[10c]

Tricyclo[3.3.1.0^{3,7}]nonane-3,7-diol (2, *n* = 1).—Prepared by the general procedure described above, the crude diol was stirred with ether and filtered to give a granular solid, mp 301–303° (lit. 297–298°), mass spectrum *m/e* 154.

Anal. Calcd for $C_9H_{14}O_2$: C, 70.10; H, 9.15. Found: C, 70.05; H, 9.19.

3,7-Dimethyltricyclo[3.3.0.0^{3,7}]octan-1-ol (4).—The crude alcohol obtained by the general procedure described above was purified by chromatography on silica gel (25 g/g). Elution with 7:3 pentane–ether removed the ketone 5 from the column. The alcohol 4 eluted with 1:1 pentane–ether. Sublimation at 55–60° (15 mm) gave a 50% yield of pure alcohol, mp 108–109°, mass spectrum *m/e* 152.

Anal. Calcd for $C_{10}H_{16}O$: C, 78.90; H, 10.59. Found: C, 79.15; H, 10.68.

Tosylate of 4.—To a solution of 1.0 g (6.5 mmol) of the alcohol in 5 ml of dry pyridine at 0° was added 1.4 g (7.2 mmol) of tosyl chloride in 5 ml of pyridine. The solution was stirred for 16 hr, after which 0.5 ml of H_2O was added and the solution was stirred for an additional hour. Most of the pyridine was removed under reduced pressure, and the residue was taken up in CH_2Cl_2 and washed with 15 ml of 1 *M* HCl, 15 ml of H_2O, and 15 ml of 10% $NaHCO_3$. The CH_2Cl_2 was evaporated under reduced pressure to give 1.6 g (80% yield) of spectroscopically pure tosylate. Recrystallization from pentane afforded an analytical sample, mp 75.5–76.5°.

Anal. Calcd for $C_{17}H_{22}O_3S$: C, 66.65; H, 7.24; S, 10.46. Found: C, 66.57; H, 7.15; S, 10.54.

1,5-Dimethylbicyclo[3.3.0]octan-3-one (5).—A solution of 152 mg (1 mmol) of 4 and 168 mg (1.5 mmol) of potassium *t*-butyl alcohol was refluxed for 4 hr. After cooling it was diluted with 7 ml of H_2O, neutralized with 1 *M* HCl, and extracted with three 20-ml portions of ether. The ether extracts were washed with two 10-ml portions of water and with saturated brine, and dried over $MgSO_4$. Evaporation of the solvent gave 130 mg (85%) of the ketone 5 (ir 1740 cm^{-1}), pure by nmr [δ 1.06 (s, 6 H), 1.73 (s, 6 H), and 2.20 (d, 4 H)]. An analytical sample was prepared by sublimation.

Anal. Calcd for $C_{10}H_{16}O$: C, 78.90; H, 10.59. Found: C, 78.65: H, 10.45.

Registry No.—2 (*n* = 1), 29898-26-8; 4, 32139-02-9; 4 tosylate, 32256-06-7; 5, 32139-03-0; bicyclo[3.3.0]-octane-1,5-diol, 32139-04-1.

Acknowledgment.—We wish to thank the donors of the Petroleum Research Fund, administered by the American Chemical Society, and the Research Corporation for partial support of this work. We also wish to thank Messrs. Richard Weinberg and Conrad Schmidt for some experimental assistance.

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[22] S. Yamamura and Y. Hirata, *J. Chem. Soc. C*, 2887 (1968).

[23] Prepared by washing the resin with 10% aqueous pyridine followed by distilled water and then methanol.

Synthesis of 1-Methyladamantano[1,2-*b*]pyrrolidine, a Novel Heterocyclic System

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Received May 24, 1971

The interesting chemistry of adamantane[1] and the biological activity of aminoadamantane and its de-

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