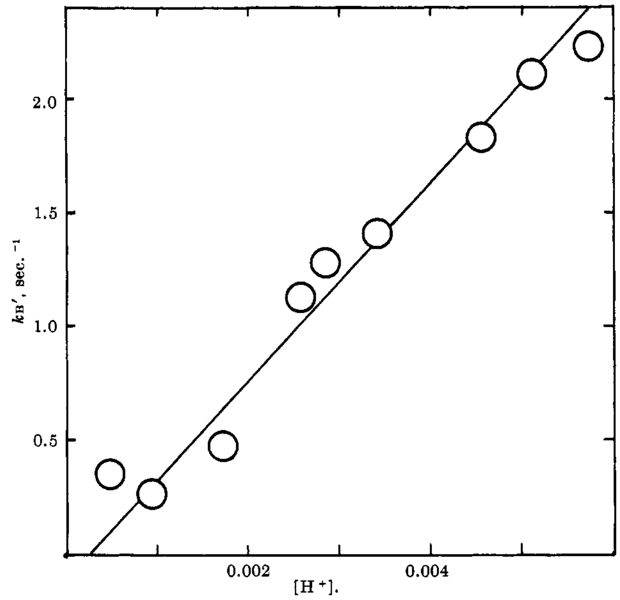

Figure 1.—Plot of k_B' calculated from the data of Table I using eq. 4 *vs.* [H$^+$].

will hence be denoted k_B'. Since V_B values were not so reproducible as desired they were measured by reference to a standard, *t*-butyl alcohol, added in known concentrations (*cf.* CVK[9]) so that we actually used eq. 4,

$$k_B' = \frac{V_B^a/V_r^a - V_B/V_r}{T_{2B}^a\, V_B/V_r} \qquad (4)$$

were the V_r terms are the heights of the reference peaks. Plots of the values of k_B' thus obtained *vs.* the hydrogen ion concentration gave a straight line from as low a hydrogen ion concentration as could be obtained (without added base) to about 0.006 *M*, after which the slope decreased. The data obtained in solutions containing more than 0.006 *M* acid were neglected since the decrease in slope is presumably due to the overlap of one peak with another, a factor not allowed for in the equation used.

To assess the validity of the assumptions that led to eq. 1, the rate constants were calculated analogously using CVM's complete eq. 3. The values thus obtained differed from those obtained using the simpler equation by less than 1%.

A plot of a set of data obtained at 35° is shown in Figure 1. From the slope of the line shown a second-order rate constant of 438 M^{-1} sec.$^{-1}$ may be calculated for the acid-catalyzed hydration of isobutyraldehyde in aqueous solution. Combination of this value with those obtained in several other such plots led to an average k_{H^+} value of 440 ± 40 M^{-1} sec.$^{-1}$.

Discussion

The rate constant we have obtained for the acid-catalyzed hydration of isobutyraldehyde in aqueous solution at 35° (440 M^{-1} sec.$^{-1}$) is larger than that obtained at 25° by Gruen and McTigue (370 M^{-1} sec.$^{-1}$)[3] using a different method. However our value is about 50% smaller than the value at 35° that would be calculated from the rate constant of Gruen and McTigue at 25° and an activation energy (16.2 kcal./mole) equal to that which may be calculated for the hydration of acetaldehyde.[5,6] We have no explanation for this discrepancy but note that the rate constant for the acid-catalyzed hydration of acetaldehyde at 25° reported by Bell, Rand, and Wynne-Jones[6] is 31% smaller than that reported by Gruen and McTigue.

Inasmuch as isobutyraldehyde is 30% hydrated at equilibrium in aqueous solution at 35°,[8] a value of 1.0 × 10^3 M^{-1} sec.$^{-1}$ may be calculated for the second-order rate constant for the acid-catalyzed dehydration of isobutyraldehyde hydrate.

Experimental

The n.m.r. spectra of isobutyraldehyde and its aqueous solutions were described earlier.[8,14] In typical kinetic experiments 0.50-ml. samples of a 3% aqueous isobutyraldehyde solution were added to each of a number of n.m.r. tubes containing 0.25 ml. of aqueous perchloric acid of known concentration under nitrogen. The n.m.r. spectrum of each sample was determined in the vicinity of the absorption due to the methyl groups at a sweep width of 50 cycles and a sweep time of 500 sec. The spectra thus obtained were not changed significantly when the sweep time was changed to 250 sec., showing that the slow sweep assumption is valid. The results obtained in a typical run are shown in Table I. There was no significant difference in the rate constants obtained at a different radiofrequency field strength; therefore, apparently the results are not complicated by saturation effects.

Table I

N.M.R. Measurements on Aqueous Isobutyraldehyde[a]

[H$^+$] × 10^4	V_B	V_r	k_B'
4.74	20.35	15.81	0.35
9.33	19.80	14.85	0.26
17.17	17.00	13.80	0.47
25.8	16.50	16.75	1.13
28.5	14.90	15.80	1.28
34.4	14.45	15.90	1.41
45.8	13.02	16.00	1.83
51.2	12.00	15.70	2.10
57.3	12.30	16.60	2.23

[a] The values of V_B^a, V_r^a, and T_{2B}^a are 21.20, 14.20, and 0.455, respectively.

Crude measurements on the rate of hydration of isobutyraldehyde were made by injecting small amounts of the aldehyde into water and very quickly measuring the change in absorbance at 2700 Å. using a Cary spectrophotometer, Model 14.

Acknowledgment.—We are indebted to the National Science Foundation for grants that aided in the purchase of the n.m.r. spectrometer and the ultraviolet-visible spectrophotometer, whose purchase was also made possible by a generous grant from the Charles F. Kettering Foundation. We also wish to thank Eastman Chemical Products, Inc., for the gift of the isobutyraldehyde used in this study.

[14] All n.m.r. spectra were determined using a Varian-A-60 instrument.

Acetylation of Triptycene[1]

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Although many derivatives of triptycene (9,10-dihydro-9,10-*o*-benzenoanthracene) have become

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[1] Supported by a grant from Smith Kline and French Laboratories, Philadelphia, Pa. We are grateful to this company for their support of this work.

known, they have been prepared by one of two routes or their combinations: the condensation of anthracene or substituted anthracenes with benzyne(s)[2] or quinone(s),[5] and the further substitution or reactions of derivatives of triptycene which had been formed by synthesis of the entire ring system.[25] The direct electrophilic substitution of triptycene has not been recorded. We have now studied the Friedel–Crafts substitution of the hydrocarbon and have found that it orients the acyl group into the 2-position.

Acetylation of triptycene in tetrachloroethane yielded 96.5% of 2-acetyltriptycene; in carbon disulfide the yield of the same ketone was much lower, and an oily ketonic substance contaminated the main reaction product. Proof for the position of the acetyl group was obtained by treating 2-lithium triptycene, from synthetic 2-bromotriptycene,[3] with acetonitrile and hydrolyzing the adduct; the resulting ketone was identical with that from the Friedel–Crafts reaction.

In measuring the n.m.r. spectrum of 2-acetyltriptycene, a mirror image quintet centered at δ 7.25 and characteristic of triptycene[29] was found. There are two doublets (δ 8.07 and 7.60) in the region of *ortho* protons and split with J_{ab} at 2 c.p.s., apparently due to *meta* splitting. A singlet at δ 5.55 is due to the bridgehead protons, while one at δ 2.45 represents the methyl protons. The ratio of these protons is 1:1:5:4:2:3. This spectrum is strikingly similar to that of 2-nitrotriptycene (see Experimental) which was synthesized from anthracene and 4-nitroanthranilic acid for spectral comparison. By contrast, the n.m.r. spectrum of synthetic 1-nitrotriptycene contains the aryl protons centered at δ 7.25 as two quintets which lack the symmetry found in unsubstituted triptycene. There are two singlets of equal intensity at δ 6.6 and 5.55; the one at δ 6.6 is evidently the bridgehead proton nearest the nitro group, and the other one the 10-bridgehead proton. The ratio of the protons in the spectrum of 1-nitrotriptycene is 6:5:1:1.

Experimental

All melting points were determined in a stirred bath and are corrected. Infrared spectra were measured with a Perkin-Elmer No. 337 spectrophotometer, and proton magnetic resonance spectra with a Varian A-60 spectrometer. Triptycene was prepared by the method of Friedman and Logullo.[3]

2-Acetyltriptycene. A.—To a stirred mixture of 23 g. (0.0905 mole) of triptycene, 250 ml. of tetrachloroethane, and 24 g. of anhydrous aluminum chloride at −20° was added 6.82 ml. (0.0905 mole) of acetyl chloride in one portion. The mixture was stirred at −20 to −30° for 30 min. and then poured into a mixture of ice and 50 ml. of 37% HCl. This mixture was extracted twice with 100 ml. of chloroform and twice with ether; the extracts were combined and dried (Na_2SO_4). After removal of the solvents under vacuum, the remaining solid was decolorized with charcoal during recrystallization from dilute ethanol; yield 25.83 g. (96.5%), m.p. 200–201°. An analytical sample had m.p. 200.5–201°; infrared bands at 2935 (w) ($>$CH), 1675 (s) (C=O), and 740 and 625 cm.$^{-1}$ (s) (aromatic H).

Anal. Calcd. for $C_{22}H_{16}O$: C, 89.16; H, 5.44. Found: C, 88.95; H, 5.52.

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[2] The following derivatives of triptycene have been prepared by this method: 2-methyl, 9-formyl, 9-bromo, 9-carbomethoxy,[3] and other 9-substituted derivatives.[4]

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[5] The following have been prepared by these methods: 9-bromo,[6-9] 9-chloro,[9] 9-iodo,[10] 9-nitro,[9,11] 9-carboxy,[8,10] 9-formyl,[10] 9-methoxy,[9] 9-methyl,[9] 9-phenoxy,[9] 9-cyano,[9] 1,4-diamino,[12] 1,4-diamino-9-carboxy,[10] 9-bromo-1,4-diamino,[7] 1,4-dibenzamido,[13] 1-benzenesulfonamido-4-hydroxy,[14,15] 9-bromo-10-methyl,[9] 9-chloro-10-methyl,[9] 9,10-dibromo,[9] 9,10-dimethyl[9]; the following derivatives of **1,4-dihydroxytriptycene**[12,16-18]: 2-methyl,[18] 9-formyl,[7] 9-carboxy,[10] 9-carbomethoxy,[10] 9-bromo,[7] 9-cyano,[9] 9-bromo-10-methyl,[9] 9,10-dimethyl,[9] 9-carboxy-10-methyl,[19] 9-carboxy,[19] 9-(α-hydroxyethyl),[9] 9-chloro,[9] 9-chloro-10-methyl,[9] 9-chloromethyl,[9] 9,10-dibromo,[9] 9-methyl,[9] 9-nitro,[9] 9-phenoxy,[9] 2-*t*-butyl,[20] 2-*t*-amyl,[20] 9-bromo-10-carbomethoxy,[19] 9-carbomethoxy,[19] 9-carboxy-10-methyl,[19] 5-carboxy,[21] 5-carboxy 1,4-diacetate,[21] 6-carboxy,[21] 6-carboxy 1,4-diacetate[21]; the following **1,4-dihydrotriptycene** derivatives: 1,4-dioxo,[9,12,17,22] 1,4-dioxime,[12] 1,4-dioxo-9-formyl,[7] 9-carboxy-1,4-dioxo,[10] 9-bromo-1,4-dioxo,[7] 9-cyano-1,4-dihydroxy,[9] 9-cyano-1,4-dioxo,[9] 9-acetyl-1,4-dioxo,[9] 9-bromo-1,4-dioxo,[9] 9-bromo-10-methyl-1,4-dioxo,[9] 9-chloromethyl-1,4-dioxo,[9] 9-chloro-1,4-dioxo,[9] 9-chloro-10-methyl-1,4-dioxo,[9] 9,10-dibromo-1,4-dioxo,[9] 9,10-dimethyl-1,4-dioxo,[9] 9-methoxy-1,4-dioxo,[9] 9-methyl-1,4-dioxo,[9] 9-nitro-1,4-dioxo,[9] 9-phenoxy-1,4-dioxo,[9] 9-bromo-10-carboxy-1,4-dioxo,[19] 9-carboxy-1,4-dioxo,[10,19] 9-carbomethoxy-1,4-dioxo,[19] 9-carboxy-10-methyl-1,4-dioxo,[19] 9-carbomethoxy-10-methyl-1,4-dioxo,[19] 9-amino-1,4-dioxo,[28] 9-[(2-hydroxy-1-naphthyl)azo]-1,4-dioxo[28]; the following **1,4,4a,9a-tetrahydrotriptycenes:** 9-bromo-1,4-dihydroxy,[9] 9-bromo-10-methyl-1,4-dihydroxy,[9] 9-chloro-1,4-dihydroxy,[9] 9-chloro-10-methyl-1,4-dihydroxy,[9] 9,10-dibromo-1,4-dihydroxy,[9] 9-ethyl-1,4-dihydroxy and 1,4-diacetate,[9] 1,4-dihydroxy,[9] 9-methoxy-1,4-dihydroxy,[9] 9-methyl-1,4-dihydroxy,[9] 9-nitro-1,4-dihydroxy,[9] 9-phenoxy-1,4-dihydroxy,[9] 1,4-dioxo,[9,16] 1-hydroxy-4-oxo,[9] 9-bromo-10-carboxy-1,4-dioxo,[19] 9-bromo-10-carbomethoxy-1,4-dioxo,[19] 9-carboxy-10-methyl-1,4-dioxo,[19] 9-carbomethoxy-10-methyl-1,4-dioxo,[19] 5-carboxy-1,4-dioxo,[21] 5-carbomethoxy-1,4-dioxo,[21] 6-carboxy-1,4-dioxo,[19] 6-carbethoxy-1,4-dioxo,[21] 9,10-dimethyl-1,4-dihydroxy,[9] 9-bromo-1,4-dioxo,[7] 9-formyl-1,4-dioxo,[7] 9-carboxy-1,4-dioxo,[7] 1,4-bis(benzoylimino),[13] 1,4-bis(benzenesulfonimino),[24] 1-benzenesulfonimino-4-oxo.[14]

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[25] The following compounds have been prepared *via* other triptycene derivatives: 1-chloro,[12] 9-chloro,[11] 9-hydroxy,[10,11,26] 9-acetoxy,[11] 9-nitroso,[11] 9-phenylazo,[11] 9-hydroxylamino,[11] 9-(N-methylhydroxylamino),[11] 9-benzalamino and N-oxide,[11] 9-azoditriptycene,[11] 9-cyano-x-methyl,[11] 9-carboxamide,[10] 9-carbonyl chloride,[10] 9-carboxazide,[10] 9-isocyanato,[10] 9-carboxy anhydride,[10] 9-hydroxymethyl,[10] 9-carboxy,[8] 9-carbomethoxy,[8] 9-carboxy-1-hydroxy (and methyl ester and lactone),[10] 9-carboxy- and 9-carbomethoxy-1,4-diacetamido,[10] 1,4-diacetamido,[12] 1,4-dibromo,[12] 1,4-dihydroxy-1,2,3,4,5,6-hexahydro and diacetate,[12] 1,4-dihydroxytetradecahydro,[12] 1,4-bis(benzenesulfonamido),[15] 1,4-bis(methanesulfonamido),[15] 1,4-diol dibenzenesulfonate,[9] 1,4-diol-1,2,3,4,4a,9a-hexahydro and 1,4-dibenzoate,[9] 9-(1-hydroxyethyl)-1,4-dihydroxy,[9] 1,4-dihydro-1-hydroxy-4-oxo,[9] 2-anilino-3-chloro-1,4-dihydro-1,4-dioxo,[27] 2-chloro-1,4-dihydro-1,4-dioxo-3-(β-hydroxyethylamino),[27] 1,4-dihydro-1,4-dioxo-2-oxy-3-pyridinium betaine,[27] 2-chloro-1,4-dihydro-1,4-dioxo-3-(β-naphthylamino),[28] 9-(chlorodiphenylmethyl),[26] 9-(hydroxydiphenylmethyl),[26] 9-triphenylboro,[26] bis(9-triptycenyl)mercury,[26] 9-phenylazo,[26] 2-anilino-3-chloro-1,4-dihydro-1,4-dioxo,[16] 2,3-dibromo- and 2,3-dichloro-1,4-dihydro-1,4-dioxo,[16] 9-carbomethoxy-1,4-diamino,[10] 9-carbomethoxy,[10] 9-methylamino,[11] 2-chloro-1,4-dioxo-3-*p*-toluidyl,[27] 9-carbomethoxy-1,4-dioxo-1,4,4a,9a-tetrahydro,[10,19] 9-triptycyl-9-triptoate,[10] 1H-6,10b-*o*-benzeno-6H-anthra[1,9-*bc*]furan-5-ol, 2a,5,5a,10c-tetrahydro,[9] 1H-6,10b-*o*-benzeno-6H-anthra[1,9-*bc*]furan,[10] bis(9-triptycenyl)selenide and diselenide.[26]

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[29] The n.m.r. spectrum of triptycene displays an A_2B_2 pattern at δ 7.15 resulting from the aromatic protons; a singlet at δ 5.3 is attributable to the two bridgehead protons. The ratio of the protons is 6:6:2.

The **2,4-dinitrophenylhydrazone** was prepared in the usual manner, washed with methanol and water, and dried over P_2O_5 at 55° for 6 hr. The crude material melted at 271–272°, and recrystallization from 2-propanol raised the melting point to 297°.

Anal. Calcd. for $C_{28}H_{20}N_4O_4$: C, 70.58; H, 4.23. Found: C, 70.34; H, 4.03.

Mixture melting points with the 2,4-DNP derivatives of the solid ketone prepared by method B, and from 2-lithium triptycene and acetonitrile (method C), respectively, showed no depression. The infrared spectra of the respective 2,4-DNP derivatives [3290 (NH), 2930 (s) (triptycene >CH), 1620 (s) (—C═N—) (s), 1595 and 1345 (s) (NO_2), 832 (s) (1,2,4-trisubstituted benzene), and 740 cm.$^{-1}$ (s) (*ortho*-disubstituted benzene)] were superimposable.

Likewise, the 4-nitrophenylhydrazone of the ketone was prepared and recrystallized from dilute ethanol; m.p. 269–270°. A mixture melting point with the 4-nitrophenylhydrazone from C was undepressed, and the infrared spectra of the two derivatives [3325 (w) (NH), 2930 (s) (triptycene >CH), 1610 (—C═N—), 1595 and 1335 (s) (NO_2), and 745 cm.$^{-1}$ (s) (*ortho*-disubstituted benzene)] were superimposable.

Anal. Calcd. for $C_{28}H_{21}N_3O_2$: C, 77.94; H, 4.91. Found: C, 78.11; H, 5.20.

B.—To 6.35 g. (0.025 mole) of triptycene in 60 ml. of carbon disulfide was added 6.67 g. (0.05 mole) of anhydrous $AlCl_3$. The mixture turned orange, then brown. With stirring, 2.55 ml. (0.025 mole) of acetic anhydride was added dropwise at a rate so as to maintain reflux (25 min.). After refluxing for 20 min. the mixture was worked up with ice–hydrochloric acid as under A and extracted into methylene chloride. The pale yellow solid was recrystallized from cyclohexane to yield 4 g. of solid, m.p. 172–183°. Thin layer chromatography (silica gel G, 8:2 methylcyclohexane–ethyl acetate, iodine vapor visualization) gave two spots, R_f 0.15 and 0.31 besides a faint spot at R_f 0.58 (triptycene). Column chromatography of 350 mg. over Florisil using methylcyclohexane–ethyl acetate (8:2) furnished two ketonic products. Both had infrared bands at 2935 (w) (>CH), 1675 (aryl conjugated ketone), and 745 cm.$^{-1}$ (*ortho*-disubstituted benzene). On standing at 4° for 1 week the material with R_f 0.31 crystallized, m.p. 201–202°; it showed no melting point depression with the ketone obtained by method A.

The oily substance (R_f 0.15) yielded an orange 2,4-DNP derivative, R_f 0.13, m.p. 298° (from isopropyl alcohol). A mixture melting point with the 2,4-DNP derivative (m.p. 297°) of the ketone from method A (R_f 0.31 in the same solvent systems) was depressed (m.p. 275–285°). The infrared spectra of the two 2,4-DNP derivatives were very similar, but the small amount of this oily ketone was not investigated further.

C.—To 0.35 g. (0.05 g.-atom) of lithium in 20 ml. of ether under a nitrogen atmosphere at −10° was added 2.45 ml. (0.025 mole) of *n*-butyl bromide in 75 ml. of ether. After stirring at −10° for 2 hr., 3.33 g. (0.01 mole) of 2-bromotriptycene[2] was added in one portion. The mixture was stirred further at 0° for 3 hr., 24 ml. of acetonitrile was added, and stirring was continued at 26° for 10 hr. Decomposition of the mixture with a saturated solution of NH_4Cl, extraction with ether, drying (Na_2SO_4), and removal of the ether furnished a solid which was recrystallized from dilute ethanol. A portion (1 g.) of 2-bromotriptycene crystallized first. The mother liquors on concentration gave 800 mg. (27%) of crude ketone; infrared bands at 1675 (C═O), 2930 (triptycene), and 740 and 625 cm.$^{-1}$ (aromatic). This material still contained some 2-bromotriptycene as shown by thin layer chromatography on silica gel G in methylcyclohexane–ethyl acetate (8:2). The bromo compound on visualization with iodine vapor showed up as a pink spot while the methyl ketone appeared as a yellow spot. Separation of the ketone was effected best as the 2,4-DNP derivative which crystallized from isopropyl alcohol and after drying over P_2O_5 for 6 hr. melted at 297°. A sample of this derivative did not depress the melting point of the corresponding derivative prepared by method A. The infrared spectra of the two derivatives were superimposable. Likewise, the *p*-nitrophenylhydrazone melted at 270–271° (from dilute ethanol); the mixture melting point with the corresponding derivative from method A was 270–271°. The two infrared spectra were superimposable.

1-Nitrotriptycene.—To a stirred refluxing mixture of 17.8 g. (0.075 mole) of 98% anthracene and 150 ml. of dry acetonitrile were added simultaneously over a 4-hr. period from separate dropping funnels 13.65 g. (0.075 mole) of 6-nitroanthranilic acid in 200 ml. of acetonitrile and 21 ml. of *n*-amyl nitrite in 75 ml. of acetonitrile. The mixture was refluxed for 1 hr. after completion of addition and then the solvent was distilled and replaced by 200 ml. of xylene. Maleic anhydride (12 g.) was added, and the mixture was refluxed for 45 min. It was cooled and diluted with 300 ml. of benzene. This mixture was extracted three times with 100 ml. of 10% NaOH, the benzene extract was dried ($NaSO_4$), and the solvent was removed. The remaining solid was chromatographed on alumina moistened with petroleum ether (b.p. 30–60°) and then diluted with 3000 ml. of petroleum ether followed by 2000 ml. of benzene. A white solid which crystallized from the petroleum ether eluate on standing was filtered off, washed with methanol, and dried; m.p. 221–221.5°, yield 1.27 g. (5.6%).

Anal. Calcd. for $C_{20}H_{13}NO_2$: C, 80.25; H, 4.38. Found: C, 80.05; H, 4.31.

The infrared spectrum contained bands at 2930 (w) (>C–H, characteristic of triptycene), 1530 and 1355 (s) (NO_2), and 735 cm.$^{-1}$ (*ortho*-disubstituted benzene).

2-Nitrotriptycene was prepared essentially as was the 1-nitro isomer, starting with 71.4 g. (0.4 mole) of anthracene, 35.1 g. (0.3 mole) of *n*-amyl nitrite, 36.4 g. (0.2 mole) of 4-nitroanthranilic acid, and 49 g. (0.5 mole) of maleic anhydride. Work-up by continuous extraction with methylene chloride, and purification by chromatography over alumina with benzene, yielded 8.4 g. (14%) of a yellow solid, m.p. 269–270°. A small amount of contaminating anthraquinone was removed by sublimation at 140° (0.075 mm.). Recrystallization from methanol gave the pure material, m.p. 270–271°, yield 3.9 g. (6.5%).

Anal. Calcd. for $C_{20}H_{13}NO_2$: C, 80.25; H, 4.38. Found: C, 80.27; H, 4.41.

The infrared spectrum showed bands at 2930 (w) (>CH), 1520 and 1340 (s) (NO_2), and 745 cm.$^{-1}$ (*ortho*-disubstituted benzene, strong); n.m.r. spectrum showed a mirror image quintet centered at δ 7.10 (unsubstituted ring protons), doublets at 8.12, and one centered at 7.88 with J_{ab} of 2 c.p.s. (in the region of *ortho* protons, apparently *meta* splitting), singlet at 7.52 (probably the proton *meta* to NO_2), doublet at 5.7 (split of 3 c.p.s., probably due to a chemical shift), ratio of protons 1:1:1:4:4:2.

Synthesis of 3-Quinuclidinol by the Cyclodehydration of (4-Piperidyl)-1,2-ethanediol

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The fact that 4-β-hydroxyethylpiperidine was cyclized to quinuclidine[2] on activated alumina prompted us to carry out this type of reaction on the model (4-piperidyl)-1,2-ethanediol in hope of obtaining a new synthetic route to an azabicyclic alcohol.

The reaction scheme that was followed is depicted on p. 1332. Thus, 4-vinylpyridine (I) was oxidized by cold aqueous permanganate to (4-pyridyl)-1,2-ethanediol (II).[3] The glycol was best isolated as its hydrochloride, which was hydrogenated to the corresponding piperidyl glycol (III) hydrochloride. About 20% of 4-β-hydroxyethylpiperidine was also formed as a hydrogenolysis by-product. The glycol hydrochloride

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[1] (a) Edgewood Arsenal; (b) Temple University; (c) Deceased.

[2] S. Leonard and S. Elkin, *J. Org. Chem.*, **27**, 4635 (1962).

[3] This glycol has not been described previously, although it has been claimed [F. E. Cislak, U. S. Patent 2,743,277 (1956)] in unspecified yield from the reaction of the N-oxide of 4-(β-hydroxyethyl)pyridine with acetic anhydride. However, this reaction could not be performed successfully in the Chemical Research and Development Laboratories, nor in those of one of our contractors.