vice versa, was found to be possible at any temperature between 0° and the boiling point of the higher boiling component.

Dialkyl sulfoxide was oxidized to the sulfone by combining it with nitric acid of any desired concentration at any temperature between 0° and the boiling point of the higher boiling component, and heating at atmospheric pressure until cessation of red fumes signified completion of the reaction.

The various oxidations are summarized in Table I.

CHEMICAL PRODUCTS DIVISION
CROWN ZELLERBACH CORPORATION
CAMAS, WASH.

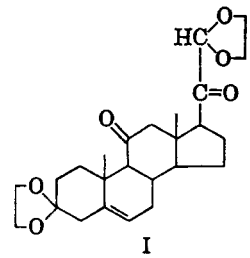
Steroidal Cyclic Ketals. XXII.[1] By-Products of the Ketalization of Cortisone and 11-Epihydrocortisone

SEYMOUR BERNSTEIN, MILTON HELLER, AND WILLIAM S. ALLEN

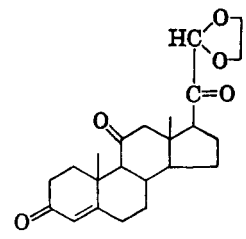
Recieved June 16, 1960

In view of the recent comment of Evans and co-workers[2] that a nonhydroxylic by-product was formed in substantial yield during the ketalization of 5α-dihydrocortisone, we wish to disclose certain similar experiences in the preparation of cortisone 3,20-bisethylene ketal[3] and 11-epihydrocortisone 3,20-bisethylene ketal.[4]

The combined mother liquors for several preparations of cortisone-3,20-bisethylene ketal[3] were evaporated and after crystallization from ethanol and then acetone, gave a new compound (I) whose elemental analyses indicated an empirical formula of $C_{25}H_{34}O_6$. There was no selective ultraviolet absorption. The infrared absorption spectrum, however, indicated the presence of two carbonyl functions, at least one ethylene ketal moiety, and a Δ^5-double bond, but no hydroxyl function. Treatment of I with sulfuric acid in methanol gave a new compound II, which obviously, from the ultraviolet and infrared absorption spectra and elemental analyses, was the result of removal of the 3-ethylene ketal group to form a Δ^4-3-one. This latter compound, moreover, still did not contain a hydroxyl group. The most convenient rationale for the by-product I was to assume it was the product of ordinary ketalization at the C3-one with a concomitant Mattox rearrangement[5] of the side chain. Since the only alcohol present in the reaction was ethylene glycol, this would necessitate that the final formulation of I would be 3,21-bisethylenedioxy-5-pregnene-11,20-dione, while II would be 21-ethylenedioxy-4-pregnene-3,11,20-trione (II).



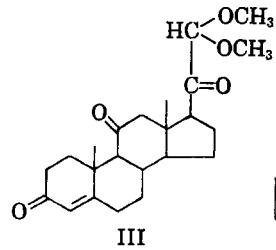
I



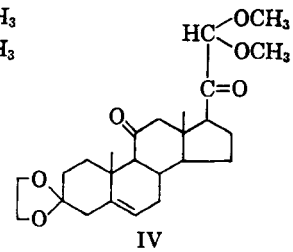
II

It was interesting to note the difficulty in removing the C21-ketal group in I which may be ascribed to the influence of the C11-carbonyl group. A similar influence has been previously found in the hydrolysis of a C20-ketal group in an 11-ketosteroid.[3,6-8] Also, the infrared absorption spectrum of I revealed the presence of carbonyl functions at 1712 and 1738 cm.$^{-1}$. The former band has been assigned to the C11-carbonyl group.[9] The assignment of the 1738 cm.$^{-1}$ band to the C20-carbonyl function implies that the band is markedly displaced from the normal 1706–1710 cm.$^{-1}$ region associated with such a group.[9,10] It would appear, then, that there is some interaction between the C20-carbonyl and the C21-ethylenedioxy groups analogous to the interaction in the 21-acetoxy-20-ketosteroids.[10]

It was thought that further confirmation of the postulated structure for I might be achieved by ketalization of 21,21-dimethoxy-4-pregnene-3,11,20-trione (III)[5] with an exchange reaction taking place at C21. Apparently no exchange reaction occurred at C21 since a new compound was obtained, presumably, 3-ethylenedioxy-21,21-dimethoxy-5-pregnene-11,20-dione (IV).



III



IV

When the mother liquors from the preparation of 11-epihydrocortisone-3,20-bisethylene ketal[4] were

(1) Paper XXI, W. S. Allen and S. Bernstein, *J. Am. Chem. Soc.*, 78, 3223 (1956).

(2) R. M. Evans, G. F. H. Green, J. S. Hunt, A. G. Long, B. Mooney, and G. H. Phillips, *J. Chem. Soc.*, 1529 (1958).

(3) R. Antonucci, S. Bernstein, M. Heller, R. Lenhard, R. Littell, and J. H. Williams, *J. Org. Chem.*, 18, 70 (1953).

(4) W. S. Allen, S. Bernstein, and R. Littell, *J. Am. Chem. Soc.*, 76, 6116 (1954).

(5) V. R. Mattox, *J. Am. Chem. Soc.*, 74, 4340 (1952).

(6) W. S. Allen, S. Bernstein, M. Heller, and R. Littell, *J. Am. Chem. Soc.*, 77, 4784 (1954).

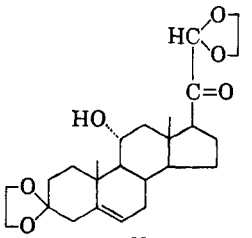
(7) J. A. Zderic and D. C. Limon, *J. Am. Chem. Soc.*, 81, 4570 (1959).

(8) K. Tsuda, N. Ikekawa, and S. Nozoe, *Chem. and Pharm. Bull.* (Tokyo), 1, 519 (1959), have recently illustrated the same sort of ketalization and Mattox rearrangement[5] while performing an exchange dioxolanation reaction on Reichstein's substance S. In this case where a C11-carbonyl group is not present, the C21-ketal group was easily hydrolyzed with dilute acid.

(9) R. N. Jones, P. Humphries, and K. Dobriner, *J. Am. Chem. Soc.*, 72, 956 (1950).

(10) R. N. Jones, V. Z. Williams, M. J. Whalen, and K. Dobriner, *J. Am. Chem. Soc.*, 70, 2024 (1948).

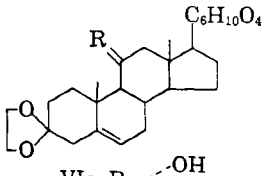
investigated, evidence for at least two by-products were obtained. Oxidation with chromium trioxide-pyridine of a crude glass isolated from the mother liquors gave in small yield compound I, indicating thereby that some 3,21-bisethylenedioxy-11α-hydroxy-5-pregnen-20-one (V) was in the glass. Direct crystallization of the crude glass from methanol, however, led to a different series of com-



V

pounds. The actual compound VIa isolated had, on the basis of elemental analyses, an empirical formula $C_{27}H_{40}O_7$. The infrared absorption spectrum showed the presence of a hydroxyl group, but no carbonyl absorption was observed. This compound could be easily acetylated to afford a derivative VIb with no hydroxyl absorption in the infrared. Furthermore, oxidation with chromic acid-pyridine (or in poorer yield with chromic acid-acetic acid) furnished a carbonyl compound VII with no hydroxyl absorption in the infrared. This set of experiments showed that the only hydroxyl grouping in VIa was most likely the 11α-ol originating from that in the starting material, 11-epihydrocortisone.

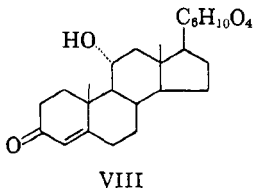
Hydrolysis of VIa with *p*-toluenesulfonic acid in acetone gave a Δ^4-3-ketone VIII, the analysis of which indicated that the side chain was unaltered in the acid treatment. It appeared to us from these series of reactions that no profound, unexplainable alterations of structure have occurred in the ring system in the synthesis of VIa from 11-epihydrocortisone. Rather, the side chain at C_{17} may be represented by the moiety, $C_6H_{10}O_4$.



VIa. R = -OH (H)

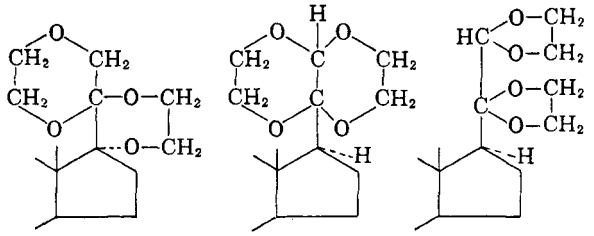
b. R = -OAc (H)

VII. R = O



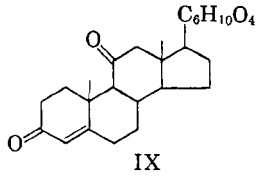
VIII

The infrared absorption spectrum of VIa indicated a high degree of 'C—O—C' absorption, but shed no light on other structural points in regard to the side chain. Hydrolysis studies also indicated the stability of the side chain toward acid. Conjecture, however, of the possible nature of the sidechain lead to the possibilities represented by *A* and *B*, formulations already proposed by Evans and co-workers.[2] The structure *A* is reminiscent of the bismethylenedioxy compounds synthesized by Beyler and associates.[11] In view of the Mattox rearrangement discussed above there is no *a priori* reason not to suggest a side chain moiety as illustrated by *C*.



A *B* *C*

In a series of experiments designed to prove rigorously the structure of the rearrangement product I, cortisone was treated at room temperature with an anhydrous hydrogen chloride solution of ethylene glycol. It was hoped that thereby compound II would be obtained. Surprisingly, a new compound IX resulted which by elemental and infrared spectral analyses appeared to contain a side chain similar to that found in VIa. Indeed, ketalization of the Δ^4-3-one group of IX by exchange dioxolanation[12] gave VII as indicated by a mixed melting point determination and by comparison of infrared spectra.



IX

A very small amount of a second product was isolated from the acidic ethylene glycol reaction on cortisone which had a very strained carbonyl absorption at 1749 cm.$^{-1}$ in its infrared spectrum. This was in addition to the expected absorption at 1711 cm.$^{-1}$ (11-carbonyl) and 1678 cm.$^{-1}$ (3-carbonyl). This was reminiscent of the spectra reported for 13α,21-epoxy-17β-methyl-18-nor-17α-4-pregnene-3,11-20-trione.[13] However, comparison with an authentic sample[13] of this compound showed the two compounds to be quite different in the fingerprint region of the infrared spectra.

In conclusion, it is felt that none of the suggested partial structures, *A*, *B* and *C*, for the side chain can be preferred in view of the method of formation and the resistance to acid hydrolysis. An attempt to hydrolyze a very small amount of VII with 60%

(11) R. E. Beyler, R. M. Moriarity, F. Hoffman, and L. H. Sarett, *J. Am. Chem. Soc.*, 80, 1517 (1958).

(12) H. J. Dauben, Jr., B. Löken, and H. J. Ringold, *J. Am. Chem. Soc.*, 76, 135 (1954).

(13) R. Hirschmann, G. A. Bailey, G. I. Poos, R. Walker, and J. M. Chemerda, *J. Am. Chem. Soc.*, 78, 4814 (1956). We wish to thank Dr. Hirschmann for the sample of 13α,21-epoxy-17β-methyl-18-nor-17α-4-pregnene-3,11,20-trione.

formic acid[11] gave a crude glass with an infrared spectrum suggesting the formation of some formates. This interesting project was then unfortunately terminated in favor of other work.

EXPERIMENTAL

Melting points. All melting points are uncorrected. *Petroleum ether.* The fraction used unless otherwise noted had a b.p. 60–70° (Skellysolve B).

3,21-Bisethylenedioxy-5-pregnene-11,20-dione (I). *A.* Mother liquors from several cortisone bisethylene ketal[3] preparations were combined and evaporated. The resultant solid was dissolved in 200 ml. of warm ethanol and allowed to stand overnight. The precipitate was filtered and crystallized from acetone to give 0.68 g. of I, m.p. 173–175°. The analytical sample had a m.p. 178–182°; ν_{max}^{Nujol} 1738, 1712 and 1100 cm.$^{-1}$; $[\alpha]_D^{25}$ +44.° (chloroform).

Anal. Calcd. for $C_{25}H_{34}O_6$ (430.52): C, 69.74; H, 7.96. Found: C, 69.78, 69.85; H, 8.17, 8.16.

B.[14] Mother liquors from a ketalization[4] of 5.0 g. of 11-epihydrocortisone were evaporated to give 1.89 g. of glass. This was dissolved in 100 ml. of pyridine and poured into a slurry of 1.27 g. of chromium trioxide in 30 ml. of pyridine. After standing for 18 hr., at room temperature the reaction mixture was poured into water and extracted with ethyl acetate. The extract was dried and evaporated to give an oil. Crystals formed after long standing of the oil dissolved in methanol. Filtration afforded 70 mg. of I, m.p. 182–184°. The infrared spectrum was identical to that of the sample prepared above in *A*.

21-Ethylenedioxy-4-pregnene-3,11,20-trione (II). A solution of 400 mg. of the 3,21-bisketal I in 40 ml. of methanol and 4.5 ml. of 8% (v/v) sulfuric acid was refluxed for 1 hr. Aqueous sodium bicarbonate was added until the solution was slightly basic. Addition of water gave a solid which was collected and dried to give 150 mg. of II, m.p. 145–151°. Recrystallization from acetone-petroleum ether raised the m.p. to 153–154°; $\lambda_{max}^{CH_3OH}$ 238 mμ (ε 15,500); ν_{max}^{Nujol} 1728, 1710, 1676 and 1622 cm.$^{-1}$; $[\alpha]_D^{25}$ +226° (chloroform).

Anal. Calcd. for $C_{23}H_{30}O_5$ (386.47): C, 71.48; H, 7.82. Found: C, 71.86, 71.60; H, 7.95, 8.18.

3-Ethylenedioxy-21,21-dimethoxy-5-pregnene-11,20-dione (IV). 21,21-Dimethoxy-4-pregnene-3,11,20-trione[5] (III, 1.0 g.) and *p*-toluenesulfonic acid (30 mg.) was added to a mixture of 35 ml. of benzene and 10 ml. of ethylene glycol. The mixture was refluxed and stirred with constant water removal for 5 hr. An additional 15 mg. of *p*-toluenesulfonic acid was added after 1 hr. A solution of potassium carbonate was added, the water layer was separated and extracted with benzene and the combined benzene extracts were evaporated. Repeated crystallization of the resultant solid from acetone-petroleum ether gave 0.415 g. of IV, m.p. 131.5–133°; ν_{max}^{Nujol} 1722 (shoulder), 1711 and 1080 cm.$^{-1}$; $[\alpha]_D^{25}$ +55° (chloroform).

Anal. Calcd. for $C_{25}H_{36}O_6$ (432.54): C, 69.42; H, 8.39. Found: C, 69.15; H, 8.57.

Isolation of compound VIa. Crystallization of the residue from the mother liquors of a preparation of 11-epihydrocortisone-3,20-bisethylene ketal gave VIa, m.p. 234–236°; ν_{max}^{Nujol} 3570 and 1100 cm.$^{-1}$; $[\alpha]_D^{25}$ −28° (chloroform).

Anal. Calcd. for $C_{27}H_{40}O_7$ (476.59): C, 68.04; H, 8.46. Found: C, 67.96; H, 8.53.

Acetylation of compound VIa (VIb). Acetylation of 500 mg. of crude VIa in 5 ml. of pyridine and 2.5 ml. of acetic anhydride in the usual manner gave 200 mg. of VIb after crystallization from acetone-petroleum ether, m.p. 233–236°; ν_{max}^{Nujol} 1730 and 1256 cm^{-1}.

Anal. Calcd. for $C_{29}H_{42}O_8$ (518.63): C, 67.16; H, 8.16; OAc, 9.54. Found: C, 67.35; H, 8.55; OAc, 9.59.

(14) This experiment was performed by R. Littell.

Compound VII. *A* A solution of 1.0 g. of VIa in 100 ml. of pyridine was treated with a slurry of 800 mg. of chromium trioxide in 140 ml. of pyridine. After the mixture was allowed to stand for 18 hr. at room temperature, 0.8 g. of sodium bicarbonate was added. The pyridine was removed by steam distillation, and the resultant mixture was extracted with chloroform. The extract was dried and evaporated to give a glass. Crystallization from ether-petroleum ether yielded 770 mg. of VII, m.p. 172–174°. The analytical sample had a m.p. 173–174°; ν_{max}^{Nujol} 1708 and 1100 cm.$^{-1}$; $[\alpha]_D^{25}$ −16° (chloroform).

Anal. Calcd. for $C_{27}H_{38}O_4$ (474.57): C, 68.33; H, 8.07. Found: C, 68.28, 68.51; H, 8.18, 8.30.

B. A solution of 0.5 g. of chromium trioxide in 20 ml. of glacial acetic acid was added to a solution of 1.0 g. of VIa in 20 ml. of acetic acid. After standing for 18 hr. at room temperature, the solution was diluted with water. The resultant solid (0.74 g.) was collected and had a m.p. 154–157°. Further crystallization from acetone-petroleum ether gave a sample of VII identical in all respects to the sample described above in *A*.

C. A solution of 0.26 g. of IX and 10 mg. of *p*-toluenesulfonic acid in 20 ml. of 2-methyl-2-ethyl-1,3-dioxolane was refluxed and slowly distilled for 5 hr. Benzene was then added to the solution and it was washed with sodium bicarbonate. Removal of the solvent afforded a glass which was submitted to chromatography on Florisil.[15] Solid was collected from the acetone-petroleum ether (23:2) eluates and had a m.p. of 170–171° with no depression of melting point on mixture with the sample obtained from *A* above. The infrared spectra of the two samples were identical.

Compound VIII. A solution of 1.0 g. of VIa and 75 mg. of *p*-toluenesulfonic acid in 75 ml. of acetone was allowed to stand at room temperature for 18 hr. Water was added and the acetone was removed under reduced pressure at room temperature. The resultant collected solid (800 mg.) had a m.p. 243–246°. Crystallization from methanol gave VIII, m.p. 258–260°; $\lambda_{max}^{C_2H_5OH}$ 241 mμ (ε 15,300); ν_{max}^{Nujol} 3700, 1672 and 1634 cm.$^{-1}$; $[\alpha]_D^{25}$ +63° (chloroform).

Anal. Calcd. for $C_{25}H_{36}O_6$ (432.54): C, 69.42; H, 8.39. Found: C, 69.61; H, 8.61.

The 3-(2,4-dinitrophenylhydrazone) of VIII prepared in the usual manner in glacial acetic acid had a m.p. 279–281°.

Anal. Calcd. for $C_{31}H_{40}O_9N_4$ (612.66): C, 60.77; H, 6.58; N, 9.15. Found: C, 60.75; H, 6.87; N, 8.53.

Compound IX. A slurry of 5.0 g. of cortisone in 40 ml. of ethylene glycol was added to 40 ml. of ethylene glycol saturated with hydrogen chloride. The mixture was stirred for 24 hr. at room temperature, and the solid (wt. 2.5 g., Fraction 1) was collected by filtration. The filtrate was diluted with water and the resultant solid was collected. Repeated crystallizations from acetone-water gave pure IX, m.p. 221.5–223°; $\lambda_{max}^{CH_3OH}$ 237–238 mμ (ε 15,500); ν_{max}^{KBr} 1710, 1682, 1627 and 1100 cm.$^{-1}$; $[\alpha]_D^{25}$ +142° (chloroform).

Anal. Calcd. for $C_{25}H_{34}O_6$ (430.52): C, 69.74; H, 7.96. Found: C, 69.82; H, 8.14.

The mother liquors from the above crystallization gave another 0.345 g. of IX, m.p. 217–219° upon partition chromatography on Celite[16] with the solvent system heptane:ethyl acetate:methanol:water (8:2:3:2).

Fraction 1 was subjected to partition chromatography on Celite[16] first with the solvent system, cyclohexane:dioxane:water (5:4:1). The second and third hold-back volumes were combined and evaporated, and again chromatographed with the solvent system heptane:ethyl acetate:methanol:

(15) Florisil is the trade-mark of the Floridin Co. for a synthetic magnesium silicate.

(16) This technique was described by S. Bernstein, M. Heller, R. Littell, S. M. Stolar, R. H. Lenhard, W. S. Allen, and I. Ringler, *J. Am. Chem. Soc.*, **81**, 1696 (1959). Celite is Johns-Manville's registered trade-mark for diatomaceous silica products.

water (8:2:3:2). Concentration of the first part of the second hold-back volume gave 0.77 g. of IX, m.p. 220–221°. Concentration of the fourth hold-back volume gave 5 mg. of an unknown compound, m.p. 175–180° (not further purified); ν_{max}^{KBr} 1749, 1711, 1678 and 1618 cm^{-1}.

Acknowledgment. We wish to thank Louis M. Brancone and associates for the analytical data, William Fulmor and associates for the spectral and optical rotational data, and Charles Pidacks and associates for the partition chromatographic separations.

ORGANIC CHEMICAL RESEARCH SECTION
LEDERLE LABORATORIES
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Dehydration of a Steroidal β-Ketol with Chromatographic Adsorbents

ROY H. BIBLE, JR., AND NORMAN W. ATWATER

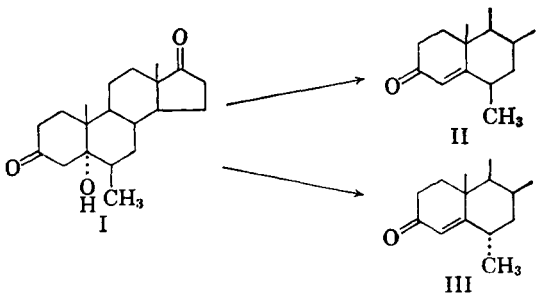
Received July 11, 1960

The preparation of steroidal 6-alkyl-Δ^4-3-ketones is usually accomplished by dehydration of the 6β-alkyl-5α-hydroxy-3-ketones which in turn arise from the action of an alkyl Grignard reagent on a 5α,6α-epoxide. A variety of basic and acidic conditions are available for the production of the 6α-alkyl derivatives[1] from this intermediate because of the great ease with which C_6 is epimerized subsequent to the dehydration step. For this same reason, however, the formation of the 6β-alkyl derivatives[2] has required careful attention to reaction conditions.

Quite by accident it was discovered that when the steroidal 5α-hydroxy-3-ketone system was chromatographed on Florisil[3] the eluates consisted of a mixture of dehydrated material along with the ketol. Further experiments in which the formation of the α,β-unsaturated ketone was followed by the appearance of its characteristic ultraviolet absorption demonstrated that the reaction was essentially complete after three hours at reflux in benzene in the presence of the Florisil. A good yield of 6β-methylandrost-4-ene-3,17-dione (II) was obtained from 6β-methyl-5α-hydroxyandrostan-3,17-dione (I)[1c] using these conditions.

This result led us then to attempt dehydration of I with the more strongly adsorbing aluminum oxide. In contrast to the previous case this reaction was quite fast and the only product isolated was 6α-methylandrost-4-en-3,17-dione (III).[4] Both procedures are characterized by the absence in the crude products of colored impurities thus allowing final crystallization with a minimum of loss.

Further examples of these methods will be described in forthcoming communications from this laboratory.



EXPERIMENTAL

6β-Methylandrost-4-ene-3,17-dione (II). A mixture of 5α-hydroxy-6β-methylandrostane-3,17-dione (I, 830 mg.),[1c] Florisil (8.3 g., 100–200 mesh), and benzene (166 ml.) was stirred at reflux temperature for 3 hr. The mixture was filtered and the solid was washed with ethyl acetate (500 ml.). Removal of the solvents from the combined filtrates followed by two recrystallizations of the resulting residue from aqueous methanol gave II; 570 mg.; m.p. 207–215°; $[\alpha]_D$ +135°; λ_{max} 241 mμ (ε 15,800). Reported[2a,5]: m.p. 207–212°; λ_{max} 242 mμ (ε 16,200); $[\alpha]_D$ +139°.

6α-Methylandrost-4-ene-3,17-dione (III). A. *By dehydration of ketol* I *with alumina.* Compound I (500 mg.) in benzene (100 ml.) was stirred at reflux temperature with basic aluminum oxide[6] (5.00 g., activity grade I) for 50 min. The mixture was filtered and the solid was washed with benzene (150 ml.). Removal of the solvent followed by one recrystallization of the resulting residue from aqueous methanol gave III; 250 mg.; m.p. 171–174°; $[\alpha]_D$ +181°. Reported[2a,5,7]: m.p. 164–167°; $[\alpha]_D$ +180°; λ_{max} 242 mμ (ε 15,650).

B. *By isomerization of the 6β-derivative* (II). A solution of Compound II (80 mg.) in ethanol (10 ml.) was refluxed with dilute sodium hydroxide (2 ml. of 0.1*N*) under nitrogen for 30 min. Dilution of the reaction mixture with water (200 ml.) containing dilute hydrochloric acid gave III; 46 mg.; m.p. 168.5–171.5°; λ_{max} 240 mμ (ε 14,520).

DIVISION OF CHEMICAL RESEARCH
G. D. SEARLE AND CO.
CHICAGO 80, ILL.

(1) (a) H. J. Ringold, J. P. Ruelas, E. Batres, and C. Djerassi, *J. Am. Chem. Soc.*, **81**, 3712 (1959); (b) A. Bowers and H. J. Ringold, *J. Am. Chem. Soc.*, **80**, 3091 (1958); (c) M. Ackroyd, W. J. Adams, B. Ellis, V. Petrow, and I. A. Stuart-Webb, *J. Chem. Soc.*, 4099 (1957); (d) M. I. Ushakov and O. S. Madaeva, *J. Gen. Chem.* (*U.S.S.R.*), **9**, 436 (1939); *Chem. Abstr.*, **33**, 9309 (1939).

(2) (a) J. A. Campbell, J. C. Babcock, and J. A. Hogg, *J. Am. Chem. Soc.*, **80**, 4717 (1958); (b) R. B. Turner, *J. Am. Chem. Soc.*, **74**, 5362 (1952).

(3) A chromatographic magnesium silicate sold by the Floridin Co., Warren, Pa. Analysis: MgO, 15.5:; SiO_2, 84%; Na_2SO_4, 0.5%.

(4) An aqueous slurry of the Florisil was at least as basic as an aqueous slurry of the aluminum oxide thus demonstrating that this result was a consequence of the action of the adsorbent itself and not of the water soluble hydroxide.

(5) Compare also Ref. 1c; H. J. Ringold, E. Batres, and G. Rosenkranz, *J. Org. Chem.*, 22, 99 (1957).

(6) A product of M. Woelm Eschwege supplied by Alupharm Chemicals.

(7) Compare also V. Grenville, D. K. Patel, V. Petrow, I. A. Stuart-Webb, and D. M. Williamson, *J. Chem. Soc.*, 4105 (1957); O. S. Madaeva, M. I. Ushakov, and N. F. Koscheleva, *J. Gen. Chem.* (*U.S.S.R.*), **10**, 213 (1940); *Chem. Abstr.*, **34**, 7292 (1940).