nan-3β,11β,17α,21-tetraol-20-one (Reichstein's substance V).[9]

Cyclization of the *dl*-diesters with potassium *t*-butoxide, followed by hydrolysis and decarboxylation, accomplished by heating with aqueous dioxane at 200–210°, gave after saponification *dl*-3β,11β-dihydroxyandrostane-17-one (VII), m.p. 249–251.5° (C, 74.4; H, 9.61), and the 13-iso compound, m.p. 216–217° (C, 74.3; H, 9.73). The diacetate of the former isomer melted at 217–217.5° (C, 70.7; H, 8.97) and had a characteristic infrared spectrum which was identical with that of authentic *d*-VII diacetate, m.p. 153.5–155°.[10,11]

It is noteworthy that when the acidic product from the ozonization in the 13-iso series was saponified to remove the acetate residues, and then isolated in the usual manner by acidification, the product was a lactonic acid, m.p. 264–266° (C, 68.4; H, 8.83; neut. equiv., 358), $\lambda_{\max}^{\text{Nujol}}$ 2.92 μ, 5.73 μ, 5.88 μ. The 5.73 μ band is indicative of a γ-lactone, and the compound is therefore represented by formula V. Since there is no doubt about the configuration at C_{13} in this compound, the formation of this lactone establishes unequivocally that the hydroxyl group at C_{11} is in the β-configuration. This observation coupled with the relationship of the C_{13} epimeric series to the natural steroids, thus constitutes conclusive confirmation of the C_{11} configuration of the natural 11-hydroxy steroids.

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[9] We are indebted to Dr. G. Rosenkranz of Syntex, S. A., for supplying us with this substance and for providing us with unpublished details for its oxidation with sodium bismuthate.

[10] M. Steiger and T. Reichstein, *Helv. Chim. Acta*, **20**, 817 (1937).

[11] We wish to thank Dr. Max Tishler of Merck and Co., Inc., for supplying us with 17α-hydroxycorticosterone from which this product was prepared.

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1,2,3,4-TETRAPHENYLFULVALENE

Sir:

Recent theoretical calculations[1,2,3,4] predicting the properties of the hitherto non-existent fulvalene hydrocarbons has stimulated interest in their synthesis. It is the purpose of this Communication to report the synthesis of the first non-fused ring fulvalene, 1,2,3,4-tetraphenylfulvalene (I).

The addition of cyclopentadienylmagnesium bromide[5] to tetracyclone in a mixture of ethyl ether and benzene afforded 74% of the almost colorless 1-cyclopentadienyltetraphenylcyclopentadiene-1-ol (II), m.p. 197.6–198.6°. Calcd. for $C_{34}H_{26}O$: C, 90.63; H, 5.82. Found: C, 90.43; H, 6.00 ($\lambda_{\max}$ 242 mμ, log ϵ 4.45; $\lambda_{\max}$ 337 mμ, log ϵ 3.85 in methanol). The infrared showed a band at 2.83 microns, but none in the region 5.6–6.1 microns indicating the presence of an hydroxyl group, no carbonyl, and thus that 1,2-addition, and not 1,4-addition, had taken place.

In refluxing bromobenzene II gave a colorless maleic anhydride adduct (III), m.p. 251–252.5° (dec.). Calcd. for $C_{38}H_{28}O_4$: C, 83.2; H, 5.1. Found: C, 82.9; H, 5.4 ($\lambda_{\max}$ 223 mμ, log ϵ 4.43; $\lambda_{\max}$ 265 mμ, log ϵ 4.08). Strong bands at 5.42 and 5.65 microns are characteristic of the anhydride ring, band at 2.90 microns indicates the hydroxyl group.

Dehydration of II was effected with iodine in boiling benzene to give the brilliant orange-red I in 27% yield, m.p. 201–202°. Calcd. for $C_{34}H_{24}$: C, 94.41; H, 5.59. Found: C, 94.04; H, 5.51 ($\lambda_{\max}$ 278 mμ, log ϵ 4.34; $\lambda_{\text{infl.}}$ 320, log ϵ 3.07; $\lambda_{\max}$ 415 mμ, log ϵ 2.95). No hydroxyl band is present in the infrared.

When III was heated at 275° for 10 minutes 98% of the theoretical quantity of water is eliminated. The red product (IV), obtained in 20% yield, melts at 177–181°. Calcd. for $C_{38}H_{26}O_3$: C, 86.01, H, 4.94. Found: C, 85.93; H, 4.6 ($\lambda_{\max}$ 240 mμ, log ϵ 4.37; $\lambda_{\text{infl.}}$ 315 mμ, log ϵ 3.91; $\lambda_{\text{infl.}}$ 415 mμ, log ϵ 2.93). There was no band at 2.90 microns; the bands at 5.42 and 5.65 microns were still present. This anhydride did not depress the melting point of the maleic anhydride adduct from I in boiling toluene in 40% yield, m.p. 178–179° (m.m.p. 178–181°). The red color of the maleic anhydride adduct indicates that the phenylated fulvene system is intact in the molecule since non-phenylated fulvenes are yellow. Thus, maleic anhydride must add to the unsubstituted C-5 ring in both I and II.

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[5] E. D. Bergmann, G. Berthier, D. Ginsburg, Y. Hirshberg, D. Lavie, S. Pinchas, B. Pullman and A. Pullman (*Bull. soc. chim. France*, 661 (1951) have reported that cyclopentadienyllithium gives no well-defined product.

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THIOESTERASES FOR ACYL AND AMINOACYL MERCAPTANS[1]

Sir:

The recognition of the function of acyl mercaptans in biosynthetic processes[2] suggests a possible role of substituted or unsubstituted α-aminoacyl mercaptans as intermediates in peptide synthesis.[3,4,5] Such a concept would be supported by the occurrence of hydrolyzing and/or transferring thioesterases with a specificity directed toward the amino acid portion of the thioester. We wish to report on the presence in ox brain cortex and liver of a group of thioesterases able to catalyze the hydroly-

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[5] H. J. Strecker, "Glutathione Symposium," Academic Press, N. Y., 1954, in press.