with two 50-ml. portions of benzene. The solvents were combined, and evaporated to one-half volume on the steam-bath. On cooling a precipitate formed which was recrystallized from alcohol. Yield was 12.5%; m.p. 151.5°.

Anal.[9] Calcd. for $C_{20}H_{22}N_2O_2$: C, 74.48; H, 6.90. Found: C, 74.30; H, 6.82.

p-Dimethylaminophenyl-6-ethoxyquinaldylcarbinol.—The above procedure was followed. The mixture was refluxed for two hours prior to the hydrolysis until the precipitate turned yellow. Yield was 24.9%; m.p. 153.6°.

Anal.[9] Calcd. for $C_{21}H_{24}N_2O_2$: C, 74.94; H, 7.21. Found: C, 74.69; H, 7.33.

Styryls.—The carbinols were dehydrated to the respective styryl derivatives by boiling in 2 *M* hydrochloric acid for one hour; the carbinol dehydrated *ca.* 99%, the 6-methoxy *ca.* 75% and the 6-ethoxy *ca.* 50%. Mixed melting points showed no depression with the corresponding styryl compounds synthesized by procedures in the literature.[10,11]

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A Stable Chloroform Adduct of 11-Epi-17α-hydroxycorticosterone

BY HELMUTH CORDS

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In view of the recent interest in Δ^4-pregnene-11α,17α,21-triol-3,20-dione,[1-7] the 11-epimer of the most important adrenal secretory product 17α-hydroxycorticosterone, we wish to describe a stable adduct of this substance with chloroform. The adduct is formed readily when the steroid is crystallized from chloroform, in which it is very difficultly soluble. It forms colorless platelets, m.p. 206–209°, $[\alpha]^{23}D$ +88 ± 2° (0.5% in ethanol) (calculated for an adduct containing one mole of chloroform: +87.8°).[8] The substance was analyzed after drying *in vacuo* (1 mm.) at 100° for two hours. *Anal.* Calcd. for $C_{21}H_{30}O_5 \cdot CHCl_3$: C, 54.83; H, 6.49; Cl, 22.08. Found: C, 54.92; H, 6.59; Cl, 21.99.

The infrared spectrum of the chloroform adduct, sampled as nujol mull, differs from that of the free 11-epi-17α-hydroxycorticosterone in that it contains a deep band at 13.28 μ, characteristic for chloroform. Moreover, the C_{21}–carbonyl band shifted from 5.83 μ for the free steroid to 5.88 μ for the adduct.

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(8) The specific rotation of the free steroid is +117° (0.5% in ethanol).

Its low solubility in chloroform, its well formed crystal shape and its stability to heat and vacuum render the chloroform adduct very suitable for purification of 11-epi-17α-hydroxycorticosterone. Microbiological synthesis of Δ^4-pregnene-11α,17α,21-triol-3,20-dione[1] generally yields slightly colored material. Recrystallization of this material from chloroform produces an almost colorless chloroform adduct (well formed platelets). The steroid can be readily freed of chloroform by crystallization from the lower alcohols, acetone or ethyl acetate.

Δ^4-Pregnene-11α,17α,21-triol-3,20-dione 11,21-diacetate[2-6,9,10] forms a similar complex with chloroform, whereas the corresponding 21-monoacetate, which was obtained in crystalline form from acetic acid–water, crystallizes from chloroform without solvate formation.

Another adduct has been observed with 5,16-pregnadiene-3β-ol-20-one and chloroform. One mole of chloroform is attached here to two moles of the steroid. This adduct, colorless platelets, is obtained by crystallization of the steroid from chloroform, and is stable to a vacuum of 1 mm., yet labile to heat. The melting point is unchanged from that of the free compound. The optical rotation, $[\alpha]^{23}D$ 20 ± 2° (0.5% in ethanol) differs, as expected, 19% from that of the free steroid. *Anal.* Calcd. for $C_{21}H_{30}O_2 \cdot {}^1/_2CHCl_3$: C, 69.02; H, 8.22; Cl, 14.22. Found: C, 69.27; H, 8.34; Cl, 14.21.

The adduct shows the strong band at 13.32 μ, characteristic for chloroform, and three additional bands at 10.54, 11.78 and 11.99 μ. The C_{20}-carbonyl shifted from 6.05 to 6.02 μ in the adduct, and four minor bands of the free steroid (8.62, 10.45, 12.41 and 12.52 μ) appeared at slightly lower wave lengths. The band at 9.86 μ is missing in the complex.

$\Delta^{5,16}$-Pregnadiene-3β-ol-20-one acetate does not form a similar adduct.

I am indebted to Dr. N. Coy for the spectrometric measurements and to Mr. J. Alicino for the microanalytical determinations.

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Phenyl Esters

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The preparations of phenyl esters have hitherto been rather tedious as they have involved the use of acid chlorides, acid anhydrides or $POCl_3$, or in the case of phenyl esters of reactive acids such as acrylic[1] or methacrylic acid,[2] somewhat circuitous synthetic routes. Phenyl esters even of reactive acids have recently been prepared with trifluoro-

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