which had precipitated, was collected and proved to be pure starting material (0.22 g.). Dilution of the filtrate with water and recrystallization of the precipitate from ether-hexane afforded 0.1 g. of colorless crystals of the ketone VIa with m.p. 153–155°, $[\alpha]^{20}\text{D}$ +33°, infrared carbonyl band at 1742 cm.$^{-1}$ typical of 17-ketosteroids.[10]

Anal. Calcd. for $C_{19}H_{28}O_2$: C, 79.12; H, 9.78. Found: C, 78.89; H, 9.79.

Δ^7-Androsten-3β-ol-17-one 3-acetate (VIb), recrystallized from dilute methanol, had m.p. 140–141°, $[\alpha]^{20}\text{D}$ +31.3°.

Anal. Calcd. for $C_{21}H_{30}O_3$: C, 76.32; H, 9.15. Found: C, 76.47; H, 9.35.

Δ^7-Androstene-3β,17β-diol (IV) (a) By Catalytic Hydrogenation of $\Delta^{5,7}$-Androstadiene-3β,17β-diol (IIa).—A solution of 0.5 g. of the dienediol IIa in 80 cc. of ethyl acetate was shaken in an atmosphere of hydrogen at room temperature and atmospheric pressure with 50 mg. of platinum oxide catalyst for three hours at which time the hydrogen uptake corresponded to approximately one mole. Filtration of the catalyst, evaporation of the solvent to dryness and recrystallization from hexane–acetone yielded 0.38 g. of colorless crystals with m.p. 181–183°, $[\alpha]^{20}\text{D}$ −23° and no selective absorption in the ultraviolet. The infrared spectrum was identical with that of a specimen prepared according to procedure (b).

Anal. Calcd. for $C_{19}H_{30}O_2$: C, 78.57; H, 10.41. Found: C, 78.22; H, 10.76.

(b) **By Lithium Aluminum Hydride Reduction of Δ^7-Androsten-3β-ol-17-one (VIa).**—The reaction was carried out in the usual manner with 3.0 g. of the ketone VIa, 60 cc. of tetrahydrofuran and 2.5 g. of lithium aluminum hydride. The diol IVa was obtained in 80% yield and possessed the same physical constants and infrared spectrum as the sample described under (a).

The diacetate IVb (ether–pentane) exhibited m.p. 119–121°, $[\alpha]^{20}\text{D}$ −28.8°.

Anal. Calcd. for $C_{23}H_{34}O_4$: C, 73.76; H, 9.15. Found: C, 73.81; H, 9.06.

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The Dipole Moments of Some Boron Trifluoride Etherates

BY ROBERT C. OSTHOFF,[1] CHARLES A. BROWN AND JANET A. HAWKINS

Since there appeared to be considerable doubt as to the magnitude of the dipole moment of $F_3BO(CH_2CH_3)_2$,[2] measurements of the electric moment of this compound were repeated. Further, it was of interest to establish the dipole moment of a higher etherate of boron trifluoride. The compound formed by boron trifluoride and tetrahydrofuran appeared to be one of the more stable etherates of boron trifluoride,[3] and its dipole moment has also been determined.

Laubengayer and Finlay[2] have measured the dipole moments of $F_3BO(CH_3)_2$, $F_3BO(CH_3)(CH_2CH_3)$ and $F_3BO(CH_2CH_3)_2$. However, these authors state that in the case of the diethyl etherate of boron trifluoride the three values of the molar orientation polarization, P_2, obtained were such as to prohibit an extrapolation to infinite dilution. The value of $P_{2\infty}$ that was used by Laubengayer and Finlay in the evaluation of the dipole moment was merely the arithmetic mean of the three values.

Experimental

Benzene.—Merck and Co., Inc., reagent grade benzene was dried over phosphorus pentoxide and distilled through a 150-cm. packed column; b.p. 80.0°, $n^{25}\text{D}$ 1.49821.

$F_3BO(CH_2CH_3)_2$.—Baker and Adamson technical boron trifluoride-diethyl etherate complex was fractionated *in vacuo;* vapor pressure at 20.0°, 2.2 mm.

Tetrahydrofuran.—du Pont "Stabilized" tetrahydrofuran was dried over calcium hydride and distilled from lithium aluminum hydride. The fraction boiling at 65–66° at 763 mm. was collected; $n^{25}\text{D}$ 1.4044.

$F_3BO(CH_2)_4$.—Matheson tank boron trifluoride was passed into the tetrahydrofuran at room temperature until the boron trifluoride was present in excess. The excess boron trifluoride was removed *in vacuo* at room temperature, and the reaction mixture was then fractionated. The fraction boiling at 69.0° and 4 mm. was collected; vapor pressure at 79.0°, 7.2 mm.

Apparatus and Methods.—Electric moments were measured in benzene solution at 25.00° using a modified heterodyne beat apparatus which will be described in a later communication.[4] The measurements were made at 100 kilocycles. The dipole moments were calculated according to the method described by Smyth.[5] The molar refraction, $R^{25}\text{D}$, of $F_3BO(CH_2CH_3)_2$ employed, was the value of 26.63 previously reported.[2] The molar refraction of $F_3BO(CH_2)_4$ was evaluated by means of a Pulfrich refractometer. In this manner the molar refraction was observed to be 28.504 cm.^{3}.

The benzene solutions that were subjected to measurements were clear and handled in the absence of air. The dielectric constant of benzene was taken as 2.273.[6]

The observed data are summarized in Table I.

TABLE I

Mole fraction, c_2	Dielectric constant, ϵ	Density, d	Total molar orientation polarization of solute, P_2
	$F_3BO(CH_2CH_3)_2$		
0.014260	2.833	0.87820	544.7
.008138	2.586	.87687	557.4
.004124	2.440	.87541	582.1
.002297	2.363	.87354	568.2
	$P_{2\infty} = 530.1$, $\mu = 4.96 \pm 0.14\ D$		
	$F_3BO(CH_2)_4$		
0.020390	3.169	0.88132	568.4
.010615	2.726	.87640	579.2
.006921	2.554	.87562	585.2
.003416	2.426	.87381	621.9
	$P_{2\infty} = 688.5$, $\mu = 5.68 \pm 0.17\ D$		

The observed dipole moment of $F_3BO(CH_2CH_3)_2$ of 4.96 ± 0.14 D was well within the experimental error of the value reported by Laubengayer and Finlay of 4.92 ± 0.38 D.

If one assumes that the dipole moment of 1.68 D[7] for the tetrahydrofuran molecule remains essentially unaltered in the addition compound, the electric moment of 5.68 D for $F_3BO(CH_2)_4$ gives a calculated bond moment for the B–O bond of 2.50 D. This value indicates a more equal sharing of the electron pair of the ether oxygen than was found in the case of $F_3BO(CH_2CH_3)_2$. This greater sharing of the donated electron pair accounts for the greater stability of the tetrahydrofuran complex. This stability increase has also been accounted for on the basis of steric concepts by Brown and Adams.[3]

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