

Anal. Calcd. for $C_{18}H_{14}ClO_4Co$: C, 55.6; H, 3.6; Co, 15.2. Found: C, 55.6; H, 3.8; Co, 15.4.

Acknowledgments.—One of the authors (P. L. P.) is grateful for financial support by the du Pont Company. This work was supported in part by the Atomic Energy Commission.

DEPARTMENT OF CHEMISTRY
HARVARD UNIVERSITY
CAMBRIDGE, MASSACHUSETTS

The Vapor Pressure of Naphthalene

BY G. W. SEARS AND E. R. HOPKE

RECEIVED JANUARY 19, 1954

Bradley and Cleasby¹ have recently reported vapor pressure data for naphthalene which were found by an effusion method. They obtained a linear $\log p$ vs. $1/T$ plot in disagreement with the measurements of Sears and Hopke,² who reported a non-linear $\log p$ vs. $1/T$ curve.

The Sears and Hopke deviation from linearity has been invalidated by the later spectroscopic detection³ of an impurity, thionaphthene, which was

- (1) R. S. Bradley and T. G. Cleasby, *J. Chem. Soc.*, 1690 (1953).
(2) G. W. Sears and E. R. Hopke, *THIS JOURNAL*, **71**, 1632 (1949).
(3) Hertha Spomer, private communication.

not removed by the purification procedures used. It has been reported independently⁴ that commercially pure naphthalene contains about 1% of thionaphthene. The non-linear vapor pressure data were measured with a Rodebush gage, which is quite sensitive to the presence of volatile impurities.

Sears and Hopke⁵ have demonstrated the volatile impurity error is minimized by the effusion method of vapor pressure measurement. The purpose of this note is to report later measurements on the vapor pressure of naphthalene by an effusion method which corroborate the data of Bradley and Cleasby.¹

From 0–20° the data obey a linear $\log p$ vs. $1/T$ relation having the same slope as reported by Bradley and Cleasby. The vapor pressures are 6% lower than those of Bradley and Cleasby. This discrepancy is rationalized in direction and magnitude as no correction was made for the orifice thickness. The orifice thickness was non-uniform and consequently no correction was attempted.

(4) W. E. Armstrong, A. B. Densham and G. Gough, *J. Chem. Soc.*, 3359 (1950).

(5) E. R. Hopke and G. W. Sears, *J. Chem. Phys.*, **19**, 1345 (1951).

GENERAL ELECTRIC RESEARCH LABORATORY
SCHENECTADY, NEW YORK

COMMUNICATIONS TO THE EDITOR

19-HYDROXY-11-DESOXYCORTICOSTERONE AND 19-HYDROXYPROGESTERONE¹

Sir:

One of the aims of work being conducted in this laboratory has been the conversion of the easily accessible cardiac aglycone strophanthidin into analogs of steroid hormones having oxygen in position 19.² Recent reports of the isolation from adrenal extracts of electrocortin, a new crystalline hormone having very pronounced sodium-retaining activity, suggest that this product may be an isomer of corticosterone.^{3,4} Therefore, we wish to report completion of the synthesis of 19-hydroxy-11-desoxycorticosterone, a new isomer of corticosterone, as well as the two possible monoacetates and the diacetate. We have also prepared 19-hydroxyprogesterone and its acetate.

Successive treatment of the sodium salt of 19-acetoxy-3-oxo- Δ^4 -etienic acid (I)⁵ with oxalyl chloride and diazomethane produced the non-crystalline 19-acetoxy-21-diazoprogesterone (II).

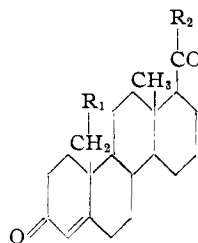
(1) This investigation was supported by a research grant from the National Cancer Institute (Grant No. CG757-C2) of the National Institutes of Health, Public Health Service.

(2) M. Ehrenstein, G. W. Barber and M. W. Gordon, *J. Org. Chem.*, **16**, 349 (1951).

(3) S. A. Simpson, J. F. Tait, A. Wettstein, R. Neher, J. von Euiw and T. Reichstein, *Experientia*, **9**, 333 (1953).

(4) V. R. Mattox, H. L. Mason and A. Albert, *Proceedings of the Staff Meetings of the Mayo Clinic*, **28**, 569 (1953); *THIS JOURNAL*, **75**, 4869 (1953).

(5) P. T. Herzig and M. Ehrenstein, *J. Org. Chem.*, **17**, 713 (1952).



- I, $R_1 = Ac$; $R_2 = OH$
 II, $R_1 = Ac$; $R_2 = CH_2N_2$
 III, $R_1 = Ac$; $R_2 = CH_2OAc$
 IV, $R_1 = Ac$; $R_2 = CH_2OH$
 V, $R_1 = H$; $R_2 = CH_2OH$
 VI, $R_1 = H$; $R_2 = CH_2N_2$
 VII, $R_1 = H$; $R_2 = CH_2OAc$
 VIII, $R_1 = H$; $R_2 = CH_3$
 IX, $R_1 = Ac$; $R_2 = CH_3$

Heating II with acetic acid gave 19-hydroxy-11-desoxycorticosterone diacetate (III), m.p. 127°, $[\alpha]^{25}_D + 210^\circ$, λ_{max}^{EtOH} 239 μ , ϵ 13,400. (*Anal.* Found: C, 70.16; H, 8.12). Hydrolysis of III with potassium bicarbonate yielded 19-acetoxy-11-desoxycorticosterone (IV), m.p. 189–190°, $[\alpha]^{25}_D + 215^\circ$, λ_{max}^{EtOH} 239 μ , ϵ 16,000. (*Anal.* Found: C, 71.06; H, 8.65.) Further hydrolysis with potassium carbonate gave 19-hydroxy-11-desoxycorticosterone (V), which melted at 163–165° after a gradual change in appearance over the range 120–145° and sintering at 153–158°, $[\alpha]^{25}_D + 180^\circ$, λ_{max}^{EtOH} 242 μ , ϵ 18,500. (*Anal.* Found: C, 72.56; H, 8.70; loss on drying, 2.44.) Hydrolysis of II with potassium bicarbonate gave 19-hydroxy-21-diazoprogesterone (VI), m.p. 166° (*Anal.* Found: C, 70.22; H, 8.21), and reaction of VI with acetic acid produced 19-hydroxy-11-desoxycorticosterone-21-acetate (VII), m.p. 197–199°, $[\alpha]^{24}_D + 178^\circ$, λ_{max}^{EtOH} 242 μ , ϵ 13,500. (*Anal.* Found: C, 70.86;