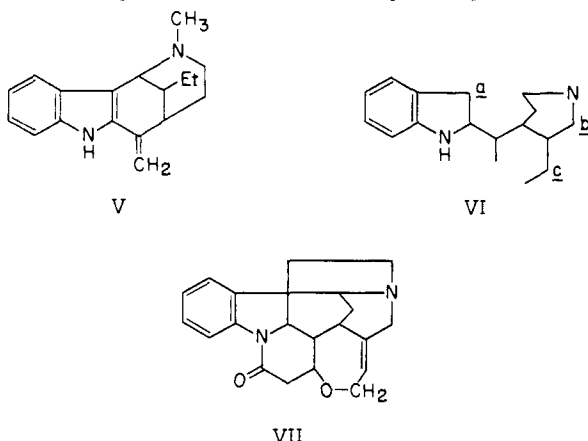


in part by chromatography on neutral alumina [activity I]. Recrystallized from methanol, the synthetic ellipticine separated in yellow prismatic needles, m.p. 312–314° [dec.] alone or in admixture with natural ellipticine. The chromatographic behavior, on neutral alumina or on paper,<sup>1</sup> and the infrared [KBr] and ultraviolet [MeOH] spectra [both unusually rich in detail] of the synthetic and natural bases were identical in all respects.

In an accompanying communication, Büchi and Warnhoff<sup>5</sup> present evidence which demonstrates that uleine, the major alkaloid of *Aspidosperma ulei* Mgf.<sup>6</sup> possesses the structure V. In view of the elaboration of N-methyltetrahydroellipticine (II) by the same plant,<sup>1,2</sup> it is of much interest that the two alkaloids possess closely related structures. The expression VI suggests a simple biogenetic relationship<sup>7</sup> between the two alkaloids [ $a \rightarrow b$ , uleine;  $a \rightarrow c$ , ellipticine] as well as



a natural connection with earlier known types [cf. strychnine (VII)].

We wish to express our appreciation to Professor George Büchi, who has kept us informed of the progress of his investigation of uleine, and to Dr. Sidney Goodwin and Professor Harold Conroy for stimulating discussions and exchanges of information. Our work has been generously supported by the Guggenheim Foundation and the National Institutes of Health.

(5) G. Büchi and E. W. Warnhoff, *THIS JOURNAL*, **81**, 4434 (1959).

(6) J. Schmutz, F. Hunziker and R. Hirt, *Helv. Chim. Acta*, **40**, 1189 (1957).

(7) R. B. Woodward, *Nature*, **162**, 155 (1948); *Angew. Chem.*, **68**, 13 (1956).

CONVERSE MEMORIAL LABORATORY  
HARVARD UNIVERSITY  
CAMBRIDGE, MASSACHUSETTS  
RESEARCH LABORATORIES  
CHAS. PFIZER AND CO., INC.  
BROOKLYN 6, NEW YORK

R. B. WOODWARD  
G. A. IACOBUCCI  
F. A. HOCHSTEIN

RECEIVED JULY 8, 1959

#### THE MOLECULAR STRUCTURE OF $(\text{Me}_2\text{N})_3(\text{BH}_2)_3$

Sir:

The compound previously reported<sup>1</sup> to be  $(\text{Me}_2\text{N})_3\text{B}_3\text{H}_4$  has been shown, by a three-dimensional X-ray diffraction study to be  $(\text{Me}_2\text{N})_3(\text{BH}_2)_3$ , a cyclic trimer of  $\text{Me}_2\text{NBH}_2$  with alternating B and N atoms in a chair configuration. Presumably this

(1) A. B. Burg, *THIS JOURNAL*, **79**, 2129 (1957).

compound is closely related to the trimer of N-methylaminoborane.<sup>2</sup>

The symmetry is orthorhombic in the space group  $\text{Pn}2_1\text{a}$ , with four molecules in a unit cell having dimensions  $a = 11.20$ ,  $b = 13.17$  and  $c = 8.07$  Å., in agreement with values obtained by J. Donohue.<sup>3</sup> Thus the symmetry is lower than that shown by the related compound<sup>4</sup>  $(\text{Me}_2\text{P})_3(\text{BH}_2)_3$ . Refinement of the structure, still in progress, has reached values<sup>5</sup> of  $R = 0.23$  and  $r = 0.16$ , with bonded distances of  $1.61 \pm 0.04$  Å. for B—N and  $1.55 \pm 0.07$  Å. for N—CH<sub>3</sub>. Methyl hydrogen atoms have not yet been included in the refinement. Values of  $R_{0kl} = 0.18$ ,  $R_{h0l} = 0.25$  and  $R_{hk0} = 0.16$  have been obtained for the three principal zones.

These results extend the inorganic-organic structural analogy of B—N compounds to include the cyclohexane type of ring, in the sense that  $\text{BH}_3\text{NH}_3$  and  $\text{C}_2\text{H}_6$ , and  $\text{B}_3\text{N}_3\text{H}_6$  and  $\text{C}_6\text{H}_6$  are pairs of analogs.

Our structure proof agrees with a concurrent, and independent study<sup>6</sup> of the hydrogen hyperfine splitting of the B<sup>11</sup> n.m.r. resonance showing a single 1:3:1 triplet strongly suggesting three equivalent  $\text{BH}_2$  groups in the molecule.

We wish to thank the Office of Naval Research and the Office of Ordnance Research for support of this research. We are indebted to Professor A. B. Burg for the sample, and to Professor J. Donohue for his preliminary X-ray diffraction results.

(2) T. C. Bissot and R. W. Parry, *ibid.*, **77**, 3481 (1956).

(3) J. Donohue, private communication.

(4) W. C. Hamilton, *Acta Crystallographica*, **8**, 199 (1955).

(5) R. E. Dickerson, P. J. Wheatley, P. A. Howell and W. N. Lipscomb, *J. Chem. Phys.*, **27**, 200 (1957).

(6) G. W. Campbell and L. Johnson, *THIS JOURNAL*, to be published.

SCHOOL OF CHEMISTRY  
UNIVERSITY OF MINNESOTA  
MINNEAPOLIS 14, MINNESOTA

LOUIS M. TREFONAS  
WILLIAM N. LIPSCOMB

RECEIVED JULY 6, 1959

#### 6-METHYL-17 $\alpha$ -ACETOXY-21-FLUORO-4,6-PREGNADIENE-3,20-DIONE. A NEW ORALLY ACTIVE PROGESTIN

Sir:

Our recent discovery of the high oral progestational activity of 21-fluoro-17 $\alpha$ -acyloxyprogesterones<sup>1</sup> coupled with the demonstrated utility of 6-methylated steroids<sup>2</sup> as progestational agents, led us to attempt the synthesis of a molecule containing both of these desirable features.

6 $\alpha$ -Methyl-17 $\alpha$ -hydroxyprogesterone (I)<sup>2b</sup> was iodinated according to the method of Ringold and Stork<sup>3</sup> using 2 moles of iodine and 9.2 moles of calcium oxide per mole of steroid. The resulting, crude iodo-compound [ $\lambda_{\text{max}}^{\text{methanol}}$  240 m $\mu$ , ( $\epsilon$  8,450), 291 m $\mu$ , ( $\epsilon$  10,500); I, 28.56%] was treated with silver fluoride<sup>4</sup> plus a small quantity of silver oxide in acetonitrile for 16 hours. The crude

(1) C. G. Bergstrom, P. B. Sollman, R. T. Nicholson and R. M. Dodson, unpublished data.

(2) (a) A. David, F. Hartley, D. R. Millson and V. Petrow, *J. Pharm. Pharmacol.*, **IX**, 929 (1957); (b) J. C. Babcock, E. S. Gutsell, M. E. Herr, J. A. Hogg, J. C. Stucki, L. E. Barnes and W. E. Dullin, *THIS JOURNAL*, **80**, 2904 (1958); (c) H. J. Ringold, E. Batres and G. Rosenkranz, *J. Org. Chem.*, **22**, 99 (1957).

(3) H. J. Ringold and G. Stork, *THIS JOURNAL*, **80**, 250 (1958).

(4) P. Tannhauser, R. J. Pratt, E. V. Jensen, *ibid.*, **78**, 2658 (1956).