

relative line intensities of the diffraction patterns shows that BaTiO<sub>3</sub> retains the cubic perovskite structure throughout the temperature range 200 to 1372°. The patterns could be indexed in the cubic system, all lines being accounted for. The unit cell dimensions listed in Table I were determined from the back-reflections using the Bradley-Jay<sup>6</sup> extrapolation method. The value of  $a_0$  at

(6) A. J. Bradley and A. H. Jay, *Proc. Phys. Soc.*, **44**, 564 (1932).

201° is in close agreement with the value  $a_0 = 4.0040 \pm 0.0005$  Kx. at 200° obtained by Megaw.<sup>1</sup> We estimate that the probable error in our values of  $a_0$  amounts to about 0.0003 Kx. unit.

**Acknowledgment.**—We wish to acknowledge the assistance of the Linde Air Products Co. in support of this investigation.

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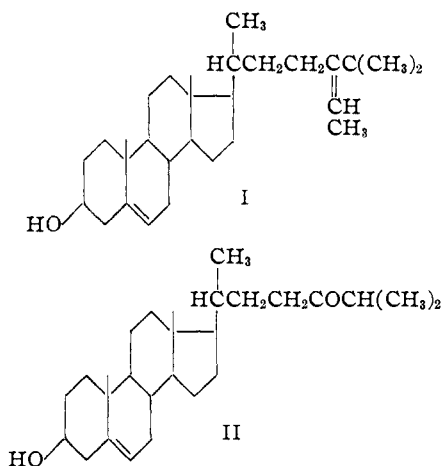
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## NOTES

### Sterols of Algae. II. The Structure of Fucosterol<sup>1</sup>

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During the past years a systematic study of the sterols of algae has been carried out in this Laboratory.<sup>2</sup> In the course of these investigations the structure for fucosterol (I) which has been proposed by MacPhillamy<sup>3</sup> has been substantiated by converting this sterol in a variety of procedures to the known 24-ketocholesterol (II).<sup>4</sup> The report on



these observations was anticipated in all its significant features by a recent British publication.<sup>5</sup> The present communication is therefore restricted to those experiments which have not already been described by the British authors.

It has been found that the conversion of I to II may readily be carried out by way of *i*-fucosteryl methyl ether. The latter was ozonized in a carbon tetrachloride solution, and the ozonide was converted by reduction with zinc in acetic acid followed by a treatment with zinc acetate into the acetate of (II).

- (1) Contributions to the Study of Marine Products. XXVII.
- (2) Bergmann and Feeney, *J. Org. Chem.*, **15**, 812 (1950).
- (3) MacPhillamy, *THIS JOURNAL*, **64**, 1732 (1942).
- (4) Riegel and Kaye, *ibid.*, **66**, 723 (1944).
- (5) Hey, Honeyman and Peal, *J. Chem. Soc.*, 2883 (1950).

### Experimental

***i*-Fucosteryl Methyl Ether.**—A solution of 1.1 g. of fucosteryl *p*-toluenesulfonate in 75 ml. of anhydrous methanol and 1.3 g. of freshly fused potassium acetate was refluxed for four hours. The solvent was then removed under reduced pressure, and the residue triturated with water and extracted with ether. The ether extract was washed with a 4% solution of sodium hydroxide and then water until neutral to litmus, dried over anhydrous potassium carbonate and evaporated to dryness under reduced pressure. The residual sirup, 0.8 g. ( $[\alpha]_D +33^\circ$ ) was dissolved in 10 ml. of hexane and shaken with 2 g. of activated alumina. After filtration and removal of the solvent there remained 0.6 g. of a sirup,  $[\alpha]^{25}_D +36.1^\circ$  ( $c$  1.0, in chloroform) which failed to yield crystalline material.

**24-Ketocholesteryl Acetate.**—A stream of oxygen containing 4.5% of ozone was passed at room temperature through a solution of 1 g. of *i*-fucosteryl methyl ether in 30 ml. of carbon tetrachloride for 20 minutes. The solvent was then removed under reduced pressure at room temperature, and the residue was dissolved in 20 ml. of glacial acetic acid. The solution was then stirred vigorously with 1 g. of zinc dust and one drop of a 1% silver nitrate solution for 15 minutes. The zinc dust was removed by centrifugation, and the solution was refluxed for two hours with 1 g. of anhydrous zinc acetate. The mixture was then diluted with water and extracted with ether, and the ether extract was washed free of acetic acid, concentrated and diluted with methanol. A crystalline material appeared which was recrystallized several times from methanol (0.4 g.), m.p. 127–128°;  $[\alpha]^{25}_D -41.1$  ( $c$  0.97, in chloroform). The m.p.'s reported for ketocholesteryl acetate are 127–128°;  $[\alpha]_D -41^\circ$ ,<sup>5</sup> and 124–131°;  $[\alpha]_D -41^\circ$ .<sup>4</sup>

*Anal.* Calcd. for C<sub>28</sub>H<sub>48</sub>O<sub>3</sub>: C, 78.75; H, 10.40. Found: C, 78.53; H, 10.50.

The 2,4-dinitrophenylhydrazone melted at 168–169°; reported<sup>6</sup> m.p. 169–170°.

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### The Synthesis of 4-Chloromethylthiazole Hydrochloride and $\beta$ -(4-Thiazolyl)-alanine Hydrochloride<sup>1</sup>

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For some time we have been engaged in preparing compounds containing the 4-thiazolylmethyl radical by syntheses which depend upon direct introduction of the latter by means of 4-chloromethylthia-

- (1) Taken from a thesis submitted by Sidney M. Fox in partial fulfillment of the requirements for the M.A. degree in June, 1947.