

The chloroacetyl-*l*-methionine was converted into glycyl-*l*-methionine by dissolving 5 g. of the compound in 20 ml. of 25% ammonium hydroxide. The solution, in a pressure flask, was heated for one hour in a water-bath maintained at 70°. The solution was then treated with silver carbonate or silver sulfate to remove the ammonium chloride. The resulting solution, after the removal of silver with hydrogen sulfide, was gently boiled with 100 mg. of Norit A and the slightly yellow filtrate was placed in a vacuum desiccator over sulfuric acid until sirupy. The glycyl-*l*-methionine was obtained as a white solid by rubbing the gummy residue with absolute alcohol. In several preparations the yield varied from 2.6 g. to 2.9 g. (57–64% yield based on the chloroacetyl-*l*-methionine used) and the melting point was 140–145°.

*Anal.* Calcd. for  $C_7H_{14}N_2O_3S$ : S, 15.54. Found: S, 15.52.

CHEMO-MEDICAL RESEARCH INSTITUTE  
GEORGETOWN UNIVERSITY  
WASHINGTON, D. C.

W. C. HESS  
M. X. SULLIVAN

RECEIVED DECEMBER 20, 1940

#### 14-Bromo-2,6,10-trimethylpentadecane

6,10,14-Trimethyl-2-pentadecanone<sup>1</sup> was prepared by ozonizing phytol. Reduction of this ketone was accomplished by dissolving 11.1 g. (0.0414 mole) of it in 100 ml. of anhydrous isopropyl alcohol and adding 6.9 g. (0.30 equiv.) of sodium, in small pieces, to the boiling mixture over a two-hour period. After the reaction mixture had been neutralized, it was extracted with ether and this extract was washed and dried. On distillation, the fraction which boiled at 146–148° at 1 mm. was collected. The yield of 6,10,14-trimethyl-2-pentadecanol, a colorless, practically odorless, mobile liquid, was 9.2 g. (82%). Although this alcohol is a new compound, it was not further characterized, but was converted directly into its bromide.

By the ordinary phosphorus tribromide method, 9.2 g. (0.0341 mole) of the above alcohol, dissolved in 80 ml. of anhydrous petroleum ether, was treated with 3.5 g.

(1) Fischer and Löwenberg, *Ann.*, **464**, 69 (1928).

(0.0129 mole) of the reagent which converted it to the bromide. Upon distillation of the reaction products, two fractions were obtained neither of which was analytically pure. The more promising fraction which weighed 3.2 g., and which represented approximately 50% of the total distillate, was dissolved in petroleum ether, extracted with concentrated sulfuric acid and redistilled. This acid treatment should have been employed before the first distillation. The 14-bromo-2,6,10-trimethylpentadecane boiled at 138–140° at 1 mm. The yield of 2.8 g. (25%) which was obtained could no doubt be substantially improved. The bromide is a bright, colorless, odorless, mobile liquid;  $n_D^{20}$  1.4614;  $d_4^{20}$  0.9726. *Anal.* Calcd. for  $C_{18}H_{37}Br$ : Br, 23.97. Found: Br, 23.75.

CHEMICAL LABORATORY  
NORTHWESTERN UNIVERSITY  
EVANSTON, ILLINOIS

PERRIN G. SMITH  
CARL E. SCHWEITZER

RECEIVED JANUARY 20, 1941

#### 2-Methyl-1,4-naphthohydroquinone Hydrogen Succinate

Incidental to some other work, 2-methyl-1,4-naphthohydroquinone hydrogen succinate was prepared, by heating 2-methyl-1,4-naphthohydroquinone with 4 moles of succinic anhydride in a bomb tube (nitrogen atmosphere) for six hours at 140°. The acidic product was then isolated via the sodium salt. Recrystallized from benzene, then from aqueous alcohol, the compound forms faintly tan-colored small prisms, melting at 176–178°. It is readily soluble in sodium bicarbonate solution.

*Anal.* Calcd. for  $C_{18}H_{14}O_5$ : C, 65.66; H, 5.15. Found: C, 65.91; H, 5.45.

The compound showed, in 2  $\gamma$  doses, a clotting time of two minutes, five hours after injection (chicks on Ansbacher diet, with a clotting time of more than sixty minutes). The same result was obtained with 1  $\gamma$  doses of 2-methyl-1,4-naphthoquinone.

THE BURROUGHS WELLCOME & Co. U. S. A.  
EXPERIMENTAL RESEARCH LABORATORIES  
TUCKAHOE, N. Y.

RICHARD BALTZLY  
JOHANNES S. BUCK

RECEIVED JANUARY 8, 1941

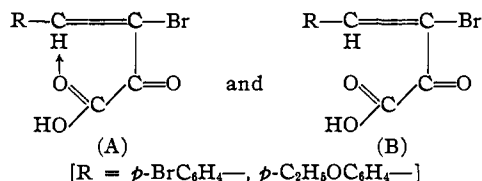
## COMMUNICATIONS TO THE EDITOR

### HYDROGEN BRIDGES AND ISOMERISM

Sir:

In recent issues of THIS JOURNAL there have appeared two articles [Reimer and Tobin, THIS JOURNAL, **62**, 2515 (1940); Reimer and Morrison, *ibid.*, **63**, 236 (1941)] in which a new type of isomerism is postulated in order to account for the existence of certain pairs of separable isomers. Although the two isomers differ markedly in their physical and chemical properties, the only difference in the structures of the two forms is stated to

be the presence of a hydrogen bridge in the one (A) and its absence in the other (B).



The separation and independent existence of isomers at ordinary temperatures implies an energy barrier amounting to at least 20 kcal.



with 12,900 for ergosterol. Too little of this product, which appears to be approximately 56% pure, was available for further purification.

The fractions which were less strongly adsorbed yielded, on systematic chromatographic analysis, a sterol acetate which appears to be homogeneous by this technique. The free sterol gave a strong Liebermann reaction and a precipitate with digitonin. The Rosenheim test was negative and no insoluble bromide could be obtained either with the free sterol or its acetate. Its composition is  $C_{29}H_{50}O$  as determined from its various derivatives: sterol, m. p. 136.5–137°,  $[\alpha]_D -41.8^\circ$ ; acetate, m. p. 137°,  $[\alpha]_D -47.6^\circ$  (calcd. for  $C_{31}H_{52}O_2$ : C, 81.5; H, 11.5. Found: C, 81.4; H, 11.6); benzoate, m. p. 137.5°,  $[\alpha]_D -17.1^\circ$  (calcd. for  $C_{36}H_{54}O_2$ : C, 83.2; H, 10.5. Found: C, 83.0; H, 10.3); and *m*-dinitrobenzoate, m. p. 200°,  $[\alpha]_D -18.3^\circ$  (calcd. for  $C_{36}H_{52}O_6N_2$ : C, 71.0; H, 8.6. Found: C, 71.2; H, 8.7).

The sterol acetate was hydrogenated in the presence of platinum oxide in glacial acetic acid. An uptake of hydrogen equivalent to one double bond was observed. The hydrogenated sterol proved to be identical with stigmastanol. Derivatives of the saturated sterol and of stigmastanol were prepared together: stanol, m. p. 134–135°,  $[\alpha]_D +23.3^\circ$ ; acetate, m. p. 129°,  $[\alpha]_D +11.5^\circ$  (calcd. for  $C_{31}H_{54}O_2$ : C, 81.2; H, 11.9. Found:

(3) All rotations were carried out in chloroform.

C, 81.1; H, 11.7); *m*-dinitrobenzoate, m. p. 210°,  $[\alpha]_D +13.9^\circ$  (calcd. for  $C_{36}H_{54}O_6N_2$ : C, 70.8; H, 8.9. Found: C, 70.7; H, 8.9); stanone, m. p. 155°,  $[\alpha]_D +38.9^\circ$ ; and the stanone oxime, m. p. 210° (calcd. for  $C_{29}H_{51}ON$ : C, 81.0; H, 12.0. Found: C, 80.8; H, 12.1). All mixed melting points with the corresponding derivatives of stigmastanol showed no depression.

The sterol is unlike any reported in sponges. The saturated sterol spongosterol<sup>4</sup> and the mono-unsaturated clionasterol<sup>5</sup> and microclionasterol,<sup>6</sup> contain 27 carbon atoms and are not well characterized.

The sterol skeleton structure is identical with that of stigmasterol. The position of the double bond is not at  $C_{5-6}$  since a comparison with 22,23-dihydrostigmasterol, synthesized by Fernholz and Ruigh,<sup>7</sup> revealed unmistakable differences.

The presence in a sponge of a sterol having the stigmasterol nucleus is of interest to comparative biochemistry. The position of the double bond in this sterol is now being studied.

(4) Henze, *Z. physiol. Chem.*, **41**, 109 (1904).

(5) Dorée, *Biochem. J.*, **4**, 72 (1909).

(6) Bergmann and Johnson, *Z. physiol. Chem.*, **222**, 220 (1933).

(7) Fernholz and Ruigh, *THIS JOURNAL*, **62**, 3346 (1940). The author is grateful to Dr. Ruigh for samples of the free sterol and its acetate.

DEPARTMENT OF BIOCHEMISTRY ABRAHAM MAZUR  
COLLEGE OF PHYSICIANS AND SURGEONS  
COLUMBIA UNIVERSITY  
NEW YORK, N. Y.

RECEIVED FEBRUARY 19, 1941

## NEW BOOKS

**Fundamentals of Semimicro Qualitative Analysis.** By ERWIN B. KELSEY and HAROLD G. DIETRICH, Assistant Professors in Chemistry, Yale University. The Macmillan Co., Inc., 60 Fifth Avenue, New York, N. Y., 1940. x + 350 pp. 12 figs. 15 × 22 cm. Price, \$2.75.

Semimicro methods in teaching chemistry have been given a wide welcome in the last few years and it is safe to conclude that they are here to stay. The time is therefore ripe for some new texts based on these methods and in the field of qualitative analysis this present book should fill the need very satisfactorily.

There are two sections of approximately equal length, entitled, respectively, "Fundamental Theory" and "Analytical Procedure." In the first we have a clear and concise discussion of the nature of solutions; salts, acids, and bases; homogeneous and heterogeneous equilibrium; complex ions; and the principles of oxidation and reduction. Both the old and newer views of ionic solutions

are presented, and considerable space is devoted to a discussion of the Brönsted-Lowry concept of acids and bases. The related concept of hydrated ions, such as  $Al(H_2O)_6^{+++}$ , as acids is discussed briefly, but the authors do not use this concept to any noticeable extent in the interpretation of experiments.

On the whole there is a fine balance between the necessarily elementary presentation and the precision and rigor of logic that ought to be the foundation of every introductory book which is to play a part in the training of scientists. Each fundamental principle is stated in words and symbols, illustrated graphically if possible, made concrete with well chosen specific examples, and clarified by the addition of actual computations with all figures included. There are practice exercises and recommendations for collateral reading.

The second section opens with a ten-page description of the special technique of semimicro analysis. The systematic procedure is then presented, in form following