

The ethereal solution, upon washing, drying, and evaporating left 1.21 g. of solid which, after recrystallization from ethanol-benzene-ligroin, yielded 0.18 g. of the phenolic ketone, m.p. 184–186°.

*Anal.*⁹ Calcd. for $C_{18}H_{20}O_2$: C, 80.58; H, 7.53. Found: C, 80.52; H, 7.83.

The acetoxy derivative was prepared in the usual manner.¹²

*Anal.*⁹ Calcd. for $C_{20}H_{22}O_2$: C, 77.41; H, 7.15. Found: C, 77.59; H, 7.54.

Attempts to Acylate I by a Friedel-Crafts Reaction. (a) **With Acetylglucyl Chloride.**—A solution of 2.00 g. of I (0.007 mole) and 1.06 g. (0.007 mole) of acetylglucyl chloride in 50 ml. of carbon disulfide was chilled to 5°. Over a period of 20 minutes 2.77 g. (0.021 mole) of anhydrous aluminum chloride was added with mechanical stirring. The mixture was allowed to come to room temperature and then heated under reflux for 5 hours. It was then cooled and poured into 40 ml. of 10% hydrochloric acid. The layers were separated, the aqueous solution was extracted twice with carbon disulfide. The combined organic layers were dried and evaporated. The residue (1.8 g.), after recrystallization from ethanol, yielded 1.66 g. of solid, m.p. 98–100°, giving no depression in a mixture melting point with I.

(b) **With Chloroacetyl Chloride.**—The reaction was carried out as described above. The residue (1.8 g.) remaining after the evaporation of the carbon disulfide was heated *in vacuo* to distill out unchanged I. The residual sirup, after several crystallizations from alcohol-water, yielded 0.88 g. of III, m.p. 78–80°, giving a positive Beilstein halogen test. Analysis showed 3.50% of chlorine instead of the anticipated 9.88%.

(12) Reference 11, p. 138.

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Some Amides of Tuberculostearic Acid

By DAVID A. SHIRLEY AND GUSTAV A. SCHMIDT¹

Tuberculostearic acid or 10-methyloctadecanoic acid is a naturally occurring fatty acid of unique structure isolated by Anderson and Chargaff² from the human tubercle bacillus. In an earlier paper³ we have described an improved method of synthesis of *dl*-tuberculostearic acid.

As a part of a general program of examination of certain derivatives of long chain fatty acids as anti-tubercular chemotherapeutic agents, we have introduced the *dl*-tuberculostearic acid fragment into several biologically active amines such as *p*-aminosalicylic acid and 4,4'-diaminodiphenyl sulfone.

Biological evaluation of these amides is being conducted by the Eli Lilly Co. of Indianapolis and we are grateful to Dr. R. G. Jones for arranging the tests.

We would also like to express appreciation to the Research Corporation of New York for a grant which supported this work.

Experimental⁴

***p,p'*-Bis-(10-methyloctadecanamido)-diphenyl Sulfone.**—Four grams (0.0135 mole) of *dl*-10-methyloctadecanoic acid was converted to the acid chloride by thionyl chloride as mentioned previously.³ To the acid chloride was added a solution of 1.6 g. (0.0065 mole) of *p,p'*-diaminodiphenyl sulfone in 15 ml. of pyridine. The mixture was refluxed for 4

hours, cooled and poured into 200 ml. of water. The precipitated solid was dissolved in acetone, decolorized with charcoal and recrystallized from ethanol to give 4.5 g. of product melting in the range 80–86.5°. Two additional recrystallizations from ethanol and one from a mixture of benzene and petroleum ether (b. p. 60–80°) gave 1.0 g. (20%) of the amide melting at 86.5–88°.

Anal. Calcd. for $C_{30}H_{54}N_2O_4S$: N, 3.46; C, 74.4; H, 10.2. Found: N, 3.48, 3.54; C, 74.2; H, 10.2.

***p*-(10-Methyloctadecanamido)-salicylic Acid.**—The acid chloride from 2.0 g. (0.0068 mole) of 10-methyloctadecanoic acid was added to a solution of 1.0 g. (0.0067 mole) of *p*-aminosalicylic acid in 20 ml. of pyridine. After standing 1 hour, the reaction mixture was poured into excess water and acidified with hydrochloric acid. The precipitated material solidified on standing and was separated and recrystallized once from ethanol, two times from 70% aqueous ethanol and two times from benzene to give 1.4 g. (48%) of the amide melting at 170–172°.

Anal. Calcd. for $C_{26}H_{48}NO_4$: N, 3.23; C, 72.2; H, 9.93. Found: N, 3.30; C, 72.0; H, 9.95.

1,4-Bis-(10'-methyloctadecanamido)-benzene.—The acid chloride from 2.5 g. (0.0084 mole) of 10-methyloctadecanoic acid and 0.4 g. (0.0037 mole) of *p*-phenylenediamine was treated in general accordance with the procedures used above except an overnight reflux period was used. There was obtained 1.0 g. (40%) of the diamide, m. p. 155–156°.

Anal. Calcd. for $C_{44}H_{80}N_2O_4$: N, 4.19; C, 79.1; H, 12.0. Found: N, 4.14; C, 79.1, 78.9; H, 12.1, 12.1.

4-(*p*-Nitrobenzenesulfonamido)-acetanilide.—Nine grams (0.0407 mole) of *p*-nitrobenzenesulfonyl chloride was added to a solution of 5.5 g. (0.037 mole) of *p*-aminoacetanilide in 30 ml. of anhydrous pyridine. After standing 1 hour, the mixture was poured into excess water and the precipitated solid (8.0 g.) recrystallized three times from ethanol. The product melted at 242–242.5° and weighed 6.0 g. (48%).

Anal. Calcd. for $C_{14}H_{13}N_3O_6S$: N, 12.54. Found: N, 12.60.

4-(*p*-Nitrobenzenesulfonamido)-aniline.—The acetanilide derivative above (1.6 g. or 0.0048 mole) was hydrolyzed by a two hour reflux with 30 ml. of 6*N* hydrochloric acid and 15 ml. of ethanol. The mixture was filtered and the filtrate neutralized with sodium acetate. The precipitated amine (1.1 g. or 80%) was recrystallized once from ethanol to give small plates, m. p. 201–202°.

Anal. Calcd. for $C_{12}H_{11}N_3O_4S$: N, 14.33. Found: N, 14.40.

4-*p*-Nitrobenzenesulfonamido)-1-(10'-methyloctadecanamido)-benzene.—Reaction of 1.0 g. (0.0034 mole) of the above amine with the acid chloride from 1.4 g. of 10-methyloctadecanoic acid in general accordance with the procedures used above gave 0.8 g. (42%) of the amide, m. p. 170.5–172°. The product was recrystallized four times from ethanol and once from a 1:1 mixture of benzene and hexane.

Anal. Calcd. for $C_{31}H_{47}N_3O_6S$: N, 7.35. Found: N, 7.55.

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Replacement of Vitamin B₁₂ by Desoxynucleotides in Promoting Growth of Certain Lactobacilli

By WILLIAM SHIVE, MARGARET E. SIBLEY AND LORENE L. ROGERS¹

Thymidine,^{2a,b} hypoxanthine desoxyriboside³ and other purine desoxyribosides^{4,5,6} replace vitamin

(1) Eli Lilly and Co. Post-doctorate Fellow.

(2) (a) Shive, Ravel and Eakin, *THIS JOURNAL*, **70**, 2614 (1948); (b) Wright, Skeggs and Huff, *J. Biol. Chem.*, **176**, 475 (1948).

(3) Shive, papers presented at Conference on Development and Uses of Antimetabolites, New York Acad. Sci., Feb., 1949, *Ann. N. Y. Acad. Sci.*, **52**, 1212 (1950).

(4) Kocher and Schindler, *Intern. Z. Vitaminforsch.*, **20**, 441 (1949).

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(6) Hoff-Jorgensen, *Abstr. 1st Intern. Congr. Biochem.*, 292 (Cambridge, 1949).

(1) Frederick G. Cottrell Research Fellow, 1949–1950.

(2) Anderson and Chargaff, *J. Biol. Chem.*, **85**, 77 (1929).

(3) Schmidt and Shirley, *THIS JOURNAL*, **71**, 3804 (1949).

(4) All melting points reported were taken on a Fisher melting point block and are uncorrected.