the procedure described by Rietz, Chapman and Fernandez.[1]

The tetradecyl derivative, m.p. 73°, was purified by crystallization from ethyl ether. *Anal.* Calcd. for $C_{62}H_{126}S_4$. C, 74.47; H, 12.71; S, 12.81. Found: C, 74.37; H, 12.69; S, 12.66.

The octadecyl derivative, m.p. 82°, was purified by crystallization from isopropyl ether. *Anal.* Calcd. for $C_{78}H_{158}S_4$: C, 76.52; H, 13.00; S, 10.49. Found: C, 76.53; H, 12.83; S, 10.59.

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(1) Rietz, Chapman and Fernandez, THIS JOURNAL, **70**, 3486 (1948).

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RECEIVED JUNE 9, 1952

2,2-Dimethyl-5-dimethylammonium-1,3-benzodioxole Iodide and 2,2-Dimethyl-5-trimethylammonium-1,3-benzodioxole Iodide

A solution of 6.5 g. (0.033 mole) of 2,2-dimethyl-5-nitro-1,3-benzodioxole[1] in 200 ml. of absolute ethanol was hydrogenated in the presence of 1.5 g. of Raney nickel catalyst at 60° and 5 atmospheres of hydrogen for 3 hours. The resulting amine solution was separated from catalyst by filtration in an inert atmosphere, then evaporated under reduced pressure to half its volume, and mixed with 10 g. (0.07 mole) of methyl iodide. A precipitate which formed soon after addition of the methyl iodide was isolated after four days and washed with ethyl ether. The colorless precipitate was extracted with 30 ml. of hot chloroform, and the chloroform solution evaporated to dryness. The chloroform-insoluble fraction was recrystallized from absolute ethanol; yield 0.85 g., m.p. 264° (dec.). The chloroform-soluble fraction was purified in the same way; yield 1.5 g., m.p. 216° (dec.). The analysis of the higher-melting compound corresponded to that of 2,2-dimethyl-5-trimethylammonium-1,3-benzodioxole iodide. *Anal.* Calcd. for $C_{12}H_{18}NO_2I$: C, 43.0; H, 5.4; N, 4.2. Found: C, 42.8; H, 5.6; N, 3.9. The lower-melting compound gave an analysis corresponding to that calculated for 2,2-dimethyl-5-dimethylammonium-1,3-benzodioxole iodide. *Anal.* Calcd. for $C_{11}H_{16}NO_2I$: C, 41.1; H, 5.0; N, 4.4. Found: C, 41.3; H, 5.2; N, 4.1.

Additional amounts of both products were isolated from the alcoholic mother liquor of the reaction mixture by precipitation with ether.

Attempted conversion of the two compounds to the corresponding catechol polymethylammonium chlorides by treatment with a hot mixture of aqueous hydrochloric acid and silver chloride led to the isolation of glasses which could not be crystallized. These glasses, when made basic, were subject to air oxidation, which causes them to become highly colored.

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(1) G. Slooff, *Rec. trav. chim.*, **54**, 995 (1935).

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RECEIVED JULY 7, 1952

The γ-Lactone of 3-Tridecyl-4-hydroxy-2-butenoic Acid

1-Acetoxy-2-pentadecanone.—A 70% yield of a white waxy solid, b.p. 153–163° (2 mm.), m.p. 49–52°, was obtained by treating myristoyl chloride with diazomethane and the azide thus obtained with glacial acetic acid.

3-Tridecyl-4-hydroxy-2-butenoic Acid, γ-Lactone.—An anhydrous benzene–ether solution of 1-acetoxy-2-pentadecanone (14.0 g.) reacted with granular zinc (3.53 g.) and ethyl bromoacetate (7.35 g.). The zinc complex was hydrolyzed and the intermediate thus obtained was dehydrated and lactonized by a saturated solution of anhydrous hydrogen bromide in glacial acetic acid. An oil, b.p. 185–195° (3–4 mm.), was obtained and recrystallization from alcohol and water gave a white solid, m.p. 57–58°. The product gave a positive Legal test, a negative potassium ferricyanide test, a positive Tollens test and it did not add bromine. The Legal test became negative after treatment with alcoholic potassium hydroxide. The melting point of a mixture with myristic acid was lowered 5°.

Anal. Calcd. for $C_{17}H_{30}O_2$: C, 76.6; H, 11.4. Found: C, 75.6; H, 11.4.

1-Acetoxy-2-heptadecanone.—A 68% yield of a white waxy solid, m.p. 62–63°, was obtained from palmitoyl chloride as described above. The product gave a positive ketone test with 2,4-dinitrophenylhydrazine. The melting point of a mixture with palmitic acid was not depressed. Two attempts to prepare a lactone from this compound failed.

Anal. Calcd. for $C_{19}H_{36}O_3$: C, 73.0; H, 11.6. Found: C, 73.0; H, 11.6.

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RECEIVED JUNE 9, 1952

α-(Benzylmercaptomethyl)-succinic Anhydride

Dimethyl α-(Benzylmercaptomethyl)-succinate.—A solution of 31.6 g. (0.2 mole) of dimethyl itaconate in 50 cc. of absolute ethanol was treated with 27.2 g. (0.22 mole) of benzylmercaptan after the addition of 5 drops of piperidine. The mixture was refluxed for 10 hr., then the solvent was removed and the residue heated at *ca.* 110–120° for two hours before fractionation. A 78% yield of golden oil, b.p. 153–160° (0.7–0.8 mm.) was obtained; redistillation gave pure dimethyl α-(benzylmercaptomethyl)-succinate, b.p. 154–156° (0.7 mm.), $n^{25}D$ 1.5250.

Anal. Calcd. for $C_{14}H_{18}O_4S$: S, 11.35. Found: S, 11.30.

α-(Benzylmercaptomethyl)-succinic Acid.—A mixture of 30.5 g. (0.107 mole) of dimethyl α-(benzylmercaptomethyl)-succinate and a solution of 15.5 g. of 85% potassium hydroxide (0.235 mole) in 50 cc. of water was refluxed five hours. The solution was charcoaled, extracted with ether and the aqueous layer acidified, extracted with ether and dried. A creamy white solid (21.5 g., 79% yield) remained after the removal of solvent and cooling of the residue. The crude product (m.p. 104–106°) was crystallized twice from 3:2 hexane–benzene to yield the pure acid in the form of white blades, m.p. 108.5–109° (cor.).

Anal. Calcd. for $C_{12}H_{14}O_4S$: C, 56.67; H, 5.55; S, 12.61. Found: C, 56.49; H, 5.33; S, 12.65.

α-(Benzylmercaptomethyl)-succinic Anhydride.—There was refluxed 15.26 g. (0.6 mole) of α-(benzylmercaptomethyl)-succinic acid and 50 cc. of acetic anhydride for 4 hours, and then the excess acetic anhydride removed. The residue was distilled, and 11.8 g. (83% yield) of golden oil collected at 150–155° (0.2 mm.). The oil solidified after some standing and crystallized from benzene–cyclohexane as fine white needles, m.p. 58–58.5° (cor.).

Anal. Calcd. for $C_{12}H_{12}O_3S$: C, 61.00; H, 5.12; S, 13.57. Found: C, 60.70; H, 5.36; S, 13.32.

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RECEIVED MAY 15, 1952

Ethyl β-Benzylmercaptopropionate

A solution of 12.0 g. (0.52 mole) of sodium in 150 cc. of absolute ethanol was treated (reflux, stirring, 0.5 hr.) with 64.5 g. (0.52 mole) of benzylmercaptan at 30–40°, then refluxed for an hour. To the sodium benzylmercaptide was added (one hour) a solution of 90.5 g. (0.5 mole) of ethyl β-bromopropionate in 75 cc. of absolute ethanol and the mixture refluxed three hours. The mixture was quenched in considerable water, extracted with ether, then the extracts washed (saturated sodium chloride solution) and dried over sodium sulfate. Fractionation of the extract gave 92.7 g. (87.5% yield) of ethyl β-benzylmercaptopropionate; b.p. 101–103° (0.22 mm.), $n^{25}D$ 1.5305.

Anal. Calcd. for $C_{11}H_{16}O_2S$: C, 62.23; H, 9.05; S, 15.10. Found:[1] C, 62.29; H, 9.37; S, 14.88.

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(1) Analyses by Mr. M. E. Auerbach and Miss P. Curran.