

The dimer of cyclohexane, which consists of about 80% bicyclohexyl and 20% of cyclohexyl- Δ^1 -cyclohexene, is readily dehydrogenated to biphenyl. Among the trimers of cyclohexene are products that yield *m*- and *p*-terphenyl on dehydrogenation.

From the tetramer fraction a small quantity of a solid tetramer, $C_{24}H_{38}$, that melts at 269–271°, was isolated. This tetramer was dehydrogenated

with difficulty to a mixture of at least two compounds that approximate the formula $C_{24}H_{18}$ but their properties do not correspond with any known compounds of this formula in the literature.

Fractions that contain pentamers, hexamers, and heptamers have been separated from the polymerization mixture.

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Branched-Chain Fatty Acids. III. New Method of Introducing the Branching Methyl Group. Synthesis of 15-Methyloctadecanoic Acid and 14-Methyltetracosanoic Acid

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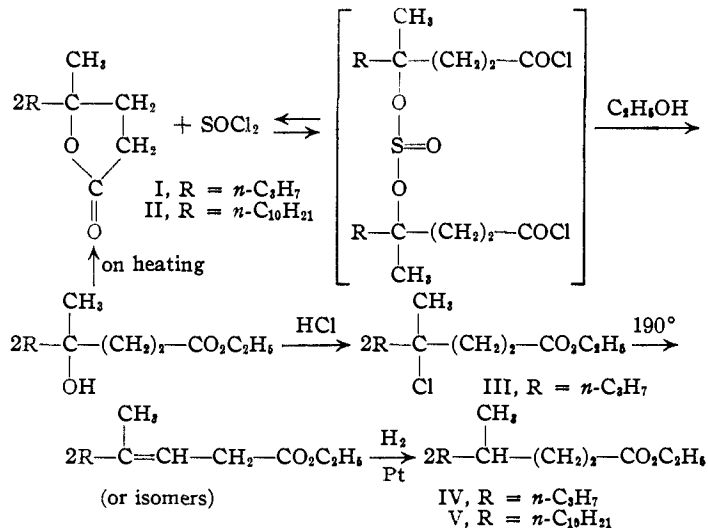
As a continuation of the program² of synthesizing branched-chain acids, 15-methyloctadecanoic acid and 14-methyltetracosanoic acid³ have been prepared by Clemmensen reduction of the esters of the corresponding 10-keto acids. The keto esters were prepared by the previously-used method² involving the reaction between a dialkylcadmium compound and ω -carbethoxynonyl chloride. The dialkylcadmium compounds were obtained from the appropriate alkyl halides by way of the Grignard reagents.

The branched-chain halide necessary for preparation of 15-methyloctadecanoic acid by this method is 1-bromo-5-methyloctane. The corresponding alcohol was initially prepared by us from 2-bromopentane by extending the chain twice by means of the reaction between a Grignard reagent and ethylene oxide. Since the 15-methyloctadecanoic acid obtained from this sample of bromide had the m. p. of 41.0–43.5° it was thought possible that this rather broad m. p. might be due to the presence of an isomeric bromide in the 2-bromopentane used as starting material. The preparation of such a bromide in a strictly pure condition is difficult if at all possible.⁴

In order to test this point, 1-bromo-5-methyloctane was prepared by a method, described in the next paragraph, which involves no branched-chain starting materials and no secondary halides. 15-Methyloctadecanoic

acid prepared from this sample of halide also melted at 41.0–43.5°. Since the crystal form of this acid appears to change during melting and since the m. p. varies somewhat, depending on how long the crystals have stood since crystallization, it is felt that the broad m. p. is due to the occurrence of polymorphic forms rather than the presence of an impurity.

The starting material for the above-mentioned preparation of a pure branched-chain alkyl halide



is a γ,γ -dialkylbutyrolactone. Such lactones have been prepared by several workers⁵ from a Grignard reagent and ethyl levulinate or levulinic acid. Yields have been reported as 35% or less. By carrying out the reaction at 0° and adding sufficient benzene to dissolve the ether-insoluble reaction product, it has been found that γ -

(1) Taken in part from the M.S. Theses of Clark E. Adams, Leonard L. Bennett, Jr., and Ulma D. Register.

(2) Preceding paper in this series, Cason and Prout, *THIS JOURNAL*, **66**, 46 (1944).

(3) Prepared for biological testing at the request of Dr. Bruno Gerstl of the Yale University School of Medicine.

(4) Sherrill, Otto and Pickett, *THIS JOURNAL*, **51**, 3027 (1929); Sherrill, Baldwin and Hass, *ibid.*, **51**, 3036 (1929); Whitmore, "Organic Chemistry," D. Van Nostrand Co., New York, N. Y., 1937, p. 73.

(5) Grignard and Moissan, *Compt. rend.*, **135**, 629 (1902); Jones and Tattersall, *J. Chem. Soc.*, **85**, 1691 (1904); Schritza, *J. Russ. Phys.-Chem. Soc.*, **44**, 1853; *Chem. Abs.*, **7**, 1174 (1913); Noyes and Marvel, *THIS JOURNAL*, **39**, 1269 (1917); Porter, *ibid.*, **45**, 1086 (1923); Frank, Arvan, Richter and Vanneman, *ibid.*, **66**, 4 (1944).

methyl- γ -propylbutyrolactone (I) may be obtained in 73% yield and γ -decyl- γ -methylbutyrolactone (II) in 84% yield.

Conversion of these lactones to the corresponding 4-methyl esters was effected according to the scheme shown in the chart.

The over-all yields in this process were 77% for ethyl 4-methylheptoate (IV) and 88% for ethyl 4-methyltetradecanoate (V). Conversion of ethyl 4-methylheptoate to the desired 1-bromo-5-methyloctane involved reduction of the ester to the corresponding alcohol and extension of the chain one carbon atom by the reaction of formaldehyde with the Grignard reagent. Reduction of ethyl 4-methyltetradecanoate and treatment of the resulting alcohol with hydrogen bromide gave 1-bromo-4-methyltetradecane, the halide necessary for synthesis of 14-methyltetraacosanoic acid.

The reaction between a lactone and thionyl chloride has previously been used by Barbier and Locquin,⁶ who obtained secondary hydroxy esters as final products, and by Plentl and Bogert,⁷ who obtained the corresponding unsaturated ester from a spiro-lactone. In the present work, when γ -methyl- γ -propylbutyrolactone was treated with thionyl chloride followed by ethanol, a small yield (15-30%) of unsaturated ester was obtained; however, the chief product of the reaction was a mixture of the original lactone and ethyl 4-chloro-4-methylheptoate. It has been found subsequently that if the alcohol used in the second stage of the reaction is saturated with hydrogen chloride an 85% yield of ethyl 4-chloro-4-methylheptoate may be obtained. This ester is not formed directly from the lactone and hydrogen chloride, under the experimental conditions used, for after two hours of refluxing with ethanol saturated with hydrogen chloride there was only about 20% conversion of the lactone to unsaturated ester and chloro-ester. Heating this chloro-ester at 190° converts it chiefly to ethyl 4-methylheptenoate; however, about 7% recyclization to the starting lactone occurs.

Contrary to the view expressed by Barbier and Locquin,⁶ our evidence suggests that one molecule of thionyl chloride reacts with two of lactone, in part at least, as indicated in the first equation above. This follows from the fact that in the case of γ -decyl- γ -methylbutyrolactone a 72% yield of unsaturated ester was obtained in a run in which only 0.6 mole equivalent of thionyl chloride was used. This reaction appears to be reversible, however, for the yield is increased by an increase in the ratio of thionyl chloride. In the most successful conversion of γ -decyl- γ -methylbutyrolactone to ethyl 4-methyltetradecanoate there was used a large excess of thionyl chloride which, on reacting with the ethanol used in the second stage of the reaction, furnished sufficient hydrogen chloride for conversion of the hydroxy ester to the chloro-ester.

(6) Barbier and Locquin, *Bull. soc. chim.*, [4] 13, 223, 229 (1913).

(7) Plentl and Bogert, *J. Org. Chem.*, 6, 677 (1941).

Experimental⁸

15-Methyloctadecanoic Acid

γ -Methyl- γ -propylbutyrolactone (I).—A Grignard reagent was prepared from 207 g. of propyl bromide (b. p. 70.7-71.0°) and 41 g. of magnesium in 600 cc. of ether. About 400 cc. of ether was distilled from the mixture, 200 cc. of benzene was added and the solution was forced under nitrogen pressure into a separatory funnel. This solution was added dropwise, during two hours, to a stirred solution of 212 g. of ethyl levulinate (b. p. 111-112° (36 mm.)) in 700 cc. of benzene. The temperature was maintained at -5 to 0°. During the addition, the reaction mixture separated into two liquid phases, but no precipitation of solid reaction complex occurred. After addition was complete the mixture was stirred an additional fifteen minutes at 0° then decomposed with ice and sulfuric acid. The aqueous phase was extracted twice with ether and the total extracts were washed with water, sodium bicarbonate solution, water and saturated sodium chloride solution, then filtered through a layer of anhydrous sodium sulfate. The residue remaining after removal of solvent was distilled through an 18-inch Vigreux type column. After a fore-run of 9.1 g. there was obtained 157.6 g. (73.3%) of γ -methyl- γ -propylbutyrolactone boiling at 128-129° (25 mm.).

Anal. Calcd. for $C_{15}H_{30}O_2$: C, 67.58; H, 9.92. Found: C, 67.58; H, 10.22.

When the above reaction was carried out at about 10° the yield was 64%. When ether alone was used as solvent a heavy precipitate rendered stirring difficult or impossible and yields of 23-35% were obtained. This latter difficulty was especially troublesome when propylmagnesium iodide was used (yield 10-12%).

Ethyl 4-Methylheptoate⁹ (IV).—A solution of 40 g. of the above lactone (I) and 100 g. (3 mole equivalents) of thionyl chloride (b. p. 76.1-76.6°) in 60 cc. of dry benzene was heated under reflux on a steam-bath for three hours. The cooled mixture was added with stirring, during fifteen minutes, to 200 cc. of absolute alcohol which had been saturated at 0° with hydrogen chloride gas. The heat of reaction caused the temperature of the mixture to rise from 12 to 24°. Stirring was continued for fifteen minutes after addition was complete, then solvent was completely removed from the mixture by distillation (final portion *in vacuo*). The residue was heated under reflux at 185-195° for one and one-half hour, hydrogen chloride being smoothly evolved. At the conclusion of the heating period the mixture was distilled through an 18-inch Podbielniak type column at 17 mm. pressure. There was obtained 38.1 g. of ethyl 4-methylheptenoate, b. p. 99.5-100.5°, 0.9 g. of intermediate and 3.2 g. of recovered lactone, b. p. 120-124°; yield of ester on basis of lactone consumed, 86.5%.

When this reaction was carried out as above except that 1 mole equivalent of thionyl chloride was used the products were 33.5 g. of unsaturated ester and 5.9 g. of lactone; with 0.6 mole equivalent of thionyl chloride the products were 26 g. of unsaturated ester and 13.6 g. of lactone. In a typical run of the same size in which 0.6 mole equivalent of thionyl chloride was used and the ethanol used in the second stage of the reaction contained no hydrogen chloride, the products were 10.7 g. of unsaturated ester and 27.4 g. of recovered lactone which contained some ethyl 4-chloro-4-methylheptoate (mixture not pyrolyzed before distillation). The presence of the latter compound was demonstrated by pyrolyzing the recovered lactone at 190° and separating, by fractional distillation, the unsaturated ester formed.

The unsaturated ester (45 g.) was hydrogenated at low pressure in 250 cc. of 95% ethanol in the presence of 0.4 g. of platinum oxide catalyst. Hydrogenation was complete in forty minutes and the yield of ethyl 4-methylheptoate, b. p. 96-97° (23 mm.), was 40 g. (89%). The unsaturated

(8) Melting points corrected; boiling points uncorrected.

(9) Burton, *Am. Chem. J.*, 3, 390 (1881-1882); Levene and Marker, *J. Biol. Chem.*, 95, 1, 153 (1932).

ester should be hydrogenated as soon as prepared, for it has been found to alter on standing.

Ethyl 4-chloro-4-methylheptoate (III) was prepared as described in the above section, and the residue remaining after removal of solvent from the reaction mixture was distilled through an 18-inch Podbielniak type column. After a fore-run of 3.2 g. there was obtained 49.2 g. (85%) of the chloro-ester, b. p. 125.5–126.5° (16 mm.). The narrow boiling range and the equivalent weight¹⁰ determination show this ester to be essentially free of lactone.

Anal. Calcd. for $C_{10}H_{19}O_2Cl$: equiv. wt., 103.3. Found: equiv. wt., 103.7, 104.1.

Treatment of γ -Methyl- γ -propylbutyrolactone with Alcoholic Hydrogen Chloride.—A solution of 20 g. of lactone (I) in 100 cc. of absolute ethanol which had been saturated with hydrogen chloride at 0° was heated under reflux for two hours. The residue remaining after distillation of solvent was fractionated through an 18-inch Podbielniak type column at 15 mm. pressure. Fractions obtained were 1.8 g., b. p. 97–119°, 19.5 g., b. p. 119–121° and 0.5 g. of residue; total recovery, 21.8 g. (theoretical yield of ethyl 4-chloro-4-methylheptoate, 29.1 g.).

5-Methyloctanol-1.^{11,12}—(A) By reduction of ethyl 4-methylheptoate with sodium and alcohol, according to Manske,¹³ there was obtained a 68% yield of 4-methylheptanol-1,^{11,12,14,16} b. p. 105–106° (33 mm.). 1-Bromo-4-methylheptane^{11,14} was prepared from the above alcohol by use of anhydrous hydrogen bromide¹⁶; b. p. 101.5–102°; yield, 80%. The Grignard reagent prepared from this bromide was treated with formaldehyde essentially as described by Noller and Adams.¹⁷ The yield of 5-methyloctanol-1, b. p. 121–124° (37 mm.), was 58%.

(B) A commercial grade of *s*-amyl alcohol (pentanol-2) was fractionated through a 3-foot packed column, and the fraction boiling at 119–119.5° was converted to 2-bromopentane in 85% yield by use of phosphorus tribromide.¹⁸ The reaction between *s*-amylmagnesium bromide and ethylene oxide was carried out essentially as described by Huston and Agett¹⁹; however, our yield of 3-methylhexanol-1,^{12,14,19,20} b. p. 94–96° (41 mm.), was only 28%. 1-Bromo-3-methylhexane,^{12,14,20} b. p. 104–105° (100 mm.), was obtained in 88% yield from the alcohol, using hydrobromic and sulfuric acids. It was converted to the Grignard reagent and this was allowed to react with ethylene oxide; yield of 5-methyloctanol-1, b. p. 101–106° (16 mm.), 65%.

1-Bromo-5-methyloctane,¹¹ b. p. 97–98° (14 mm.), was prepared from the corresponding alcohol in 75% yield, using anhydrous hydrogen bromide.¹⁶

Ethyl 10-keto-15-methyloctadecanoate was prepared from 10 g. of 1-bromo-5-methyloctane and 8.4 g. of ω -carbethoxynonyl chloride by the method which has been described for preparation of methyl 4-keto-6-methyloctanoate²; b. p. 180–195° (1–2 mm.); yield, 7.4 g. (65%). A center cut was used for analysis.

Anal. Calcd. for $C_{21}H_{40}O_3$: C, 74.07; H, 11.84. Found: C, 73.91; H, 11.91.

10-Keto-15-methyloctadecanoic acid, prepared by saponification of the ester, crystallized from petroleum ether as short blades, m. p. 43.4–47.0°, remelting at 44.5–

(10) The equivalent weight was determined by saponification. Both the ester group and the tertiary chlorine were hydrolyzed.

(11) Levene and Marker, *J. Biol. Chem.*, **103**, 299 (1933).

(12) Dorough, Glass, Gresham, Malone and Reid, *THIS JOURNAL*, **63**, 3100 (1941).

(13) Manske, *Org. Syn.*, **14**, 20 (1934).

(14) Levene and Marker, *J. Biol. Chem.*, **106**, 173 (1934).

(15) Basu, *J. Ind. Chem. Soc.*, **8**, 319 (1931); Graves, *J. Ind. Eng. Chem.*, **23**, 1381 (1931); Koller and Kandler, *Monatsh.*, **58**, 213 (1931).

(16) Ruhoff, Burnett and Reid, *Org. Syn.*, **15**, 24 (1934).

(17) Noller and Adams, *THIS JOURNAL*, **48**, 1080 (1926).

(18) Noller and Dinsmore, *Org. Syn.*, **13**, 20 (1933).

(19) Huston and Agett, *J. Org. Chem.*, **6**, 123 (1941).

(20) Dewael and Weckerling, *Bull. soc. chim. Belg.*, **33**, 495 (1924); Levene and Marker, *J. Biol. Chem.*, **91**, 77 (1931).

46.5°. Since a sample completely melted at once when placed in a bath at 45.5°, the substance appears to be polymorphic.

Anal. Calcd. for $C_{19}H_{36}O_2$: C, 73.03; H, 11.62. Found: C, 73.37; H, 11.79.

Ethyl 15-methyloctadecanoate was prepared by Clemmensen reduction of the keto ester in alcoholic solution, according to Schneider and Spielman.²¹ From 12.9 g. of keto ester there was obtained 8.8 g. (71%) of reduced ester, b. p. 170–172° (1–2 mm.) (distillation through the 18-inch Podbielniak type column).

Anal. Calcd. for $C_{21}H_{42}O_2$: C, 77.24; H, 12.96. Found: C, 77.00; H, 12.62.

15-Methyloctadecanoic acid was obtained by saponification of the ester. Crude acid obtained from 1-bromo-5-methyloctane prepared by Procedure (A) melted at 40–42°, while that obtained from bromide prepared by Procedure (B) melted at 35–37.5°. In either case pure acid was obtained after three to five crystallizations from acetone. Immediately after crystallization the m. p. of the pure acid is 40.7–42.5°, but after standing two days or longer the m. p. is 41.0–43.5°. Repeated crystallization of samples of this acid from acetone, methanol or ligroin (34–40°) failed to alter the m. p. or behavior on melting.

Anal. Calcd. for $C_{19}H_{38}O_2$: C, 76.45; H, 12.83; mol. wt., 298.5. Found: C, 76.37; H, 12.94; mol. wt., 296.3.

The amide²² crystallized from acetone as scales, m. p. 74.5–79.5°, yield 86%. After two additional crystallizations from acetone there were obtained feathery blades melting at 78.8–79.6°.

Anal. Calcd. for $C_{19}H_{39}ON$: C, 76.71; H, 13.21. Found: C, 77.07; H, 13.25.

The tribromoanilide,²² after four crystallizations from acetone, was obtained as clusters of white needles, m. p. 102.6–103.8°. This compound also appears to be polymorphic, for a sample placed in a bath at 103° melted completely at once.

Anal. Calcd. for $C_{22}H_{40}ONBr_3$: C, 49.21; H, 6.60. Found: C, 49.95; H, 6.88.

14-Methyltetracosanoic Acid

γ -Decyl- γ -methylbutyrolactone (II) was prepared essentially as described for γ -methyl- γ -propylbutyrolactone except that no ether was distilled after preparation of the Grignard reagent and there was used 1 liter of benzene per mole of ethyl levulinate. The reaction mixture remained homogeneous. From 44.2 g. of *n*-decyl bromide (b. p. 111.0–111.5° (10 mm.)) and 26 g. of ethyl levulinate there was obtained 36.4 g. (84.4%) of lactone boiling at 179–181° (5 mm.) (18-inch Podbielniak type column).

Anal. Calcd. for $C_{18}H_{36}O_2$: C, 74.94; H, 11.74. Found: C, 74.88; H, 11.49.

Ethyl 4-Methyltetracosanoate (V).—A mixture of 100 g. of lactone, II, and 148 g. of pure thionyl chloride was heated under reflux on a steam-bath for three hours. The cooled reaction mixture was added with stirring, at 25–30°, to 190 cc. of absolute alcohol. Stirring was continued for ten minutes then the solvent was boiled off at atmospheric pressure and the residue was distilled through a 3-foot Vigreux type column at 3 mm. pressure. The yield of ethyl 4-methyltetracosanoate boiling at 153–154° was 103 g. (92.7%). No lactone was recovered. Nearly the same yield was obtained when the addition to alcohol was made at 0–5° or at the boiling point of alcohol.

One run was carried out as above except that only 30 g. (0.6 mole equivalent) of thionyl chloride was used and the alcohol used in the second step contained 10% by weight of hydrogen chloride. On distillation at 3 mm. pressure there was obtained 80.2 g. (72%) of unsaturated ester, b. p. 153.5–154°, 2.9 g. of intermediate and 13.5 g. of recovered lactone, b. p. 168–170°. When no hydrogen

(21) Schneider and Spielman, *J. Biol. Chem.*, **142**, 345 (1942).

(22) Methods used for preparation of derivatives were essentially the same as those described in the previous paper of this series (ref. 2).

chloride was added to the alcohol used in the second step the yield of unsaturated ester was 50–60% and a correspondingly larger amount of lactone was recovered.

Hydrogenation of 80 g. of the unsaturated ester in 200 cc. of 95% alcohol, using 0.45 g. of platinum oxide catalyst, gave 78 g. (96.5%) of saturated ester, b. p. 147–149° (2 mm.).

Anal. Calcd. for $C_{17}H_{34}O_2$: C, 75.49; H, 12.67. Found: C, 75.38; H, 12.32.

4-Methyltetradecanol-1 was prepared by reduction of the above-described ester (V) with sodium and absolute ethanol.¹³ From 54 g. of ester there was obtained 33.9 g. (69.5%) of alcohol, b. p. 139–141° (1–2 mm.).

Anal. Calcd. for $C_{15}H_{32}O_2$: C, 78.89; H, 14.12. Found: C, 79.01; H, 13.96.

The distillation residue from the above preparation was combined with the alkaline solution from which the alcohol was extracted. By acidification of this solution and extraction with benzene there was recovered 10 g. of **4-methyltetradecanoic acid**, b. p. 170–172° (2 mm.). The distilled acid freezes at about 32°.

Anal. Calcd. for $C_{15}H_{30}O_2$: C, 74.34; H, 12.48. Found: C, 73.85; H, 12.47.

1-Bromo-4-methyltetradecane was prepared from the corresponding alcohol with anhydrous hydrogen bromide¹⁶ in 88% yield, b. p. 145–146.5° (1–2 mm.) (18-inch Vigreux type column).

Anal. Calcd. for $C_{15}H_{31}Br$: C, 61.86; H, 10.73. Found: C, 62.25; H, 10.88.

Ethyl 10-keto-14-methyltetracosanoate was prepared by the method which has been described for preparation of methyl 4-keto-7-methyloctoate.² From 15 g. of 1-bromo-4-methyltetradecane and 10.82 g. of ω -carbethoxynonyl chloride there was obtained 15.5 g. (77.8%) of the keto ester, b. p. 242–245° (1 mm.). The ester was distilled from a Claisen flask. The high carbon values obtained on analysis of this ester and the corresponding reduced ester are no doubt due to the presence of considerable quantities of 10,18-dimethyloctacosane (from coupling of the Grignard reagent).

Anal. Calcd. for $C_{27}H_{52}O_2$: C, 76.36; H, 12.34. Found: C, 77.33; H, 12.25.

In one run the crude keto ester was saponified by refluxing for two hours with 100 cc. of 5% alcoholic sodium hydroxide. The insoluble sodium salt was collected and dissolved in water. The aqueous solution, after extraction with ether, was acidified, whereupon there separated only a small precipitate which proved to be sebacic acid. The sodium salt of 10-keto-14-methyltetracosanoic acid was found to have dissolved almost entirely in the ether extract and was recovered by evaporation of this solution. The keto acid, after four crystallizations from ligroin (30–60°), melted at 60.8–61.8°.

Anal. Calcd. for $C_{26}H_{50}O_2$: C, 75.69; H, 12.20. Found: C, 76.00; H, 12.45.

Ethyl 14-methyltetracosanoate was obtained by reduction²¹ of 14.5 g. of ethyl 10-keto-14-methyltetracosanoate. The yield of material boiling at 236–238° (1–2 mm.) was 10.1 g. (71%). Distillation was through an 18-inch Podbielniak type column.

Anal. Calcd. for $C_{27}H_{54}O_2$: C, 78.76; H, 13.47. Found: C, 80.51; H, 13.06.

14-Methyltetracosanoic Acid.—The crude acid obtained by saponification of 10 g. of the ester was semi-solid at room temperature. After one crystallization from acetone there was obtained 8.0 g. (85.5%) of acid melting at 53–57°. After three additional crystallizations from acetone and three from ligroin (30–60°) the acid melted sharply at 57.8–58.5°; however, it was shown by titration (mol. wt. found, 390.0) that this product contained about 2% neutral material (10,18-dimethyloctacosane). This neutral material could not be removed by filtration of an aqueous solution of the sodium salt, but was removed by three additional crystallizations of the acid from ligroin (80–96°) and one from acetone. The yield of pure acid, which crystallized as very fine needles or blades, was 4.3 g., m. p. 57.9–58.5°.

Anal. Calcd. for $C_{26}H_{50}O_2$: C, 78.46; H, 13.17; mol. wt., 382.7. Found: C, 78.82; H, 12.99; mol. wt., 384.0.

The amide,²² after one crystallization from butanone and two from methanol, melted at 80.2–81.0°.

Anal. Calcd. for $C_{26}H_{51}ON$: C, 78.67; H, 13.47. Found: C, 79.16; H, 12.94.

The tribromoanilide,²² after two crystallizations from butanone and one from methanol, melted at 86.7–87.5°.

Anal. Calcd. for $C_{26}H_{51}ONBr_3$: C, 53.87; H, 7.58. Found: C, 54.25; H, 7.69.

Summary

There have been prepared two new branched-chain acids, 15-methyloctadecanoic acid and 14-methyltetracosanoic acid.

There has been developed a satisfactory and general method for preparing pure branched-chain esters which may serve as intermediates for preparation of pure branched-chain alkyl halides. This method involves the preparation of a γ,γ -dialkylbutyrolactone from a Grignard reagent and ethyl levulinate, followed by opening of the lactone ring with thionyl chloride.

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