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Received March 20, 1954.

Use of Thiophene in Lengthening Carbon Chains of Aliphatic Acids by five Carbon Atoms

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In connection with other works, lengthening of aliphatic acids with several carbon atoms by a simple method was of interest. Since thiophene compounds are readily available and easily desulphurated to saturated aliphatic compounds, a method using thiophene as a starting material seems to be convenient.

Starting from an aliphatic acid RCOOH , the corresponding acylthiophene $\text{RCOC}_4\text{H}_4\text{S}$ can be prepared by one of the general methods given by Hartough ¹⁻³. This acylthiophene is reduced to the corresponding alkylthiophene $\text{RCH}_2\text{C}_4\text{H}_4\text{S}$ by a modified Wolff-Kishner method ⁴. From this alkylthiophene the 5-alkyl-2-acetylthiophene $\text{RCH}_2\text{C}_4\text{H}_4\text{SCOCH}_3$ is prepared and oxidized to the 5-alkyl-2-thiophenecarboxylic acid $\text{RCH}_2\text{C}_4\text{H}_4\text{SCOOH}$ ⁵. From thiophenecarboxylic acids, sulphur has been removed by means of Raney nickel. This operation requires great amounts of Raney nickel in order to give the hydrogen required. Since much hydrogen is produced in the preparation of Raney nickel from the alloy, it might be advantageous to use the Raney alloy directly. In this way the amount of the alloy can be greatly reduced compared with the amount necessary if the Raney nickel is first prepared. Another advantage is that neither the alloy nor the nickel residue obtained by the reduction is pyrophoric, if the alloy is used.

The method is exemplified by the preparation of *n*-heptanoic acid from acetic acid.

Experimental. 2-Acetothienone was prepared according to *Organic Syntheses* ⁶, cf. Hartough and Conley ².

2-Ethylthiophene. In a 1-l round-bottomed flask fitted with a condenser set for distillation, 79.5 g of potassium hydroxide were dissolved in 560 ml of diethylene glycol, 200 ml of hydrazine hydrate (85 %) and 103 g of 2-acetothienone were added and the mixture heated until no more 2-ethylthiophene distilled over. After washing with water, drying over sodium sulphate and distilling, the yield was 78 g, b.p. 135–140°.

5-Ethyl-2-acetothienone was prepared by the same method as 2-acetothienone.

5-Ethyl-2-thiophenecarboxylic acid was prepared by a modified procedure of that given for the preparation of 2-naphthoic acid from 2-acetylnaphthalene in *Organic Syntheses* ⁶, cf. Hartough and Conley ².

Removal of sulphur from 5-ethyl-2-thiophenecarboxylic acid. 75 g of the finely ground nickel-aluminium alloy (the minimum quantity necessary) was added slowly to a solution of 75 g of sodium hydroxide in 300 ml of distilled water and 8.8 g of 5-ethyl-2-thiophenecarboxylic acid contained in a 1-l three-necked round-bottomed flask fitted with a mechanical stirrer, a reflux condenser and a stopper, surrounded by ice. The mixture was refluxed for 5 hours, filtered while warm and the catalyst washed with a small quantity of water. The combined filtrate was acidified with concentrated hydrochloric acid and extracted with benzene. The benzene layer was washed with water, dried and distilled. The yield was 5.8 g of *n*-heptanoic acid, b.p. 122–124°/14 mm. A quantity of the acid was converted into the amide, m.p. 97 (from water) and this showed no melting point depression when mixed with an authentic sample of *n*-heptamid.

The author is indebted to Messrs. Pharmacia Ltd. for permission to publish this paper and to Fil.Lic. Arne Brändström for his valuable help.

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Received March 20, 1954.