With tri-2-butylphosphine oxide no product was obtained when the ether solution was refluxed overnight before final work-up. However, when ether was displaced with dioxane and the dioxane solution refluxed overnight, a 70% yield of product was obtained.

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Some Substituted Thiophenes and 2,2′-Bithiophenes

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The properties of a number of substituted thiophenes and 2,2′-bithiophenes are reported. Per cent yields and melting or boiling points are given for the reported compounds, which were among those prepared in a study of the synthesis of carboxylic acids.

PREVIOUS work (*1*) by the author on hydrocarbons has now been successfully extended to the preparation of two substituted thiophenes and four substituted 2,2′-bithiophenes. Data are given in Tables I and II. The basic conditions used for the necessary acylations and reductions (*2*) were the same for both thiophene and bithiophene, affording a basis for comparison of yields.

Boiling points were determined during vacuum distillation of the compounds using standard apparatus. Melting points were determined using a Vanderkamp "Melt-Pointer" (Scientific Glass Apparatus Co., Inc., Catalog No. M-1945). Mixed melting points were determined using a 50 to 50 mixture of the synthetic and commercial products. Neutralization equivalents were determined using standard titration methods. Ultimate analyses for carbon, hydrogen, and sulfur were in accord with theory.

Table I. Properties of Substituted Thiophenes and 2,2′-Bithiophenes

				%C		%H		%S	
Compound	Yield	B.P., ° C.	M.P., ° C.	Found	Calcd.	Found	Calcd.	Found	Calcd.
Methyl-5-(2-*n*-butyl-5-thenoyl)valerate	74.0	188–190 (1 mm.)	34–35	63.95	63.79	7.94	7.79	11.11	11.34
6-(2-*n*-Butyl-5-thienyl)-hexanoic acid	85.5	171–174 (0.1 mm.)	...	66.35	66.09	9.09	8.72	12.68	12.60
5-*n*-Butyryl-2,2′-bithiophene	76.0	...	76–77	60.69	60.98	5.08	5.12	26.89	27.14
5-*n*-Butyl-2,2′-bithiophene	65.6	109–113 (1.5 mm.)	...	64.76	64.79	6.42	6.34	28.63	28.84
Methyl 5-[5′-*n*-butyl-5-(2,2′-bithenoyl)]-valerate	48.1	...	68–69	62.57	62.61	6.74	6.64	17.82	17.59
6-[5′-*n*-Butyl-5-(2,2′bithienyl)]-hexanoic acid	76.6	...	60–61	64.22	64.25	7.06	7.19	18.88	19.06

Table II. Properties of Carboxylic Acids

				Neut. Equiv.	
Acid	Yield	M.P., ° C.	Mixed M.P., ° C.	Found	Calcd.
Tetradecanoic	85.2	54.0	54.0	228.0	228.4
Octadecanoic	76.5	69.5	69.5	284.4	284.4

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