16-Halohexadecanoic Acids

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AS PART OF STUDY of the surface activity of long-chain alkanoic acids, a series of 16-halohexadecanoic acids was required. Since it was important that the compounds be free of branched isomers and halogen substitution other than at the terminal carbon atom, our synthetic approach was based on the sodium borohydride cleavage of the ozonide (*2*) of 16-heptadecenoic acid to give 16-hydroxyhexadecanoic acid. Under the mild conditions of this reaction, little rearrangement would be expected. The hydroxy acid was readily converted to the corresponding halogen acids (Table I) by conventional procedures.

The 16-heptadecenoic acid was prepared by the reduction of 7-oxo-16-heptadecenoic acid (*3*) in a manner similar to that reported by Brace (*1*) after completion of our work. It was shown recently by Sisido and Kawanisi (*4*) that the usual Wolff-Kishner reaction modifications result in extensive double bond migration during the reduction of unsaturated keto acids. As indicated by Brace (*1*), we also found that a shortened heating period for decomposition of the hydrazone gave the unsaturated acid without rearrangement.

Table I. 16-X-Hexadecanoic Acids

			Analysis							
			Calcd.				Found			
X	Yield %	M.P. °C.	C	H	X	Neut. Eq.	C	H	X	Neut. Eq.
OH	55	92–93	70.54	11.84	...	...	70.20	12.10	...	...
Cl[a]	74	61–61.5	66.07	10.74	12.19	290.9	66.18	10.95	11.99	288.2
Br[b]	66	69.5–70.0	57.31	9.32	23.83	335.3	57.46	9.62	23.46	333.5
I[c]	62	74–74.5	50.26	8.17	33.19	382.3	50.75	8.00	33.25	384.2

[a] From the hydroxy acid, $SOCl_2$, and pyridine. [b] From the hydroxy acid and 48% aq. HBr in acetic acid. [c] From the bromo acid and NaI; all three halogen compounds were recrystallized from light petroleum ether.

LITERATURE CITED

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Viscosity of Hydrocarbons. *n*-Butane

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THE VISCOSITY of *n*-butane has been investigated in some detail. Early measurements by Titani (*13*) provided information concerning the viscosity of *n*-butane at temperatures between 68° and 248° F. at atmospheric pressure. Swift and co-workers (*12*) reported on the viscosity of *n*-butane bubble point liquid at temperatures from 68° to 212° F. Lipkin (*5*) also reported data on the viscosity of saturated liquid *n*-butane at temperatures between −100° and 100° F. Starling *et al.* (*11*) described investigations of the viscosity of *n*-butane in the critical region including isotherms at 306°, 307°, and 309° F. Sage (*9*) investigated the effect of pressure on the viscosity of *n*-butane at pressures up to 2000 p.s.i.a. at a temperature interval between 100° and 220° F. for the liquid and gas phases. These latter data were obtained with a rolling-ball viscometer, and unexpected uncertainties exist concerning the reported viscosities in the gas phase at elevated pressures. More recently, Dolan and co-workers (*2*) reported information about the viscosity of *n*-butane at a number of temperatures in the liquid and gas phases at pressures up to 8000 p.s.i.a. at 100° F. and to 1000 p.s.i.a. at 340° F. These data were obtained with a capillary-tube viscometer (*3*).

It is believed that before confidence can be placed in viscosity measurements, data should be obtained over the range of temperatures and pressures of interest from two widely different types of instruments. With this in view measurements of the viscosity of *n*-butane were undertaken to strengthen confidence in the values reported by Dolan (*2*), whose investigation appears to be the most complete