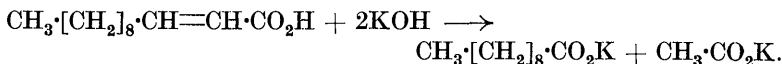


CCLXXVII.—*The Preparation of n-Decoic Acid.*

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n-DECOIC acid is difficult to obtain. Rochussen's method of preparation (Ber. Schimmel & Co. Akt. Ges., 1929, 181) from the scission of bromolauric acid esters by fusion with potassium hydroxide gives a mixture of two acids, which are separable only with difficulty.

From the decomposition of bromolauric acid chloride with various alcohols, we have obtained seven esters in the pure state. Δ^{α} -Dodecenoic acid is obtained from these by heating them with alcoholic potash. If this acid is used instead of the bromo-ester, pure *n*-decoic acid can be easily obtained :



E X P E R I M E N T A L.

α -Bromolauric Esters.—Lauric acid (m. p. 43°) was first converted into the acid chloride by thionyl chloride, and then brominated according to Guérin (*Bull. Soc. chim.*, 1903, 29, 1123); the action of the appropriate alcohol then afforded the required ester. The average yield from 100 g. of lauric acid was 120 g. The esters were purified by distillation under reduced pressure. They are colourless liquids, insoluble in water, but soluble in most organic solvents, such as chloroform, benzene, carbon tetrachloride, acetone, and alcohol.

Esters of α -bromolauric acid.

	B. p./ 3 mm.	B. p./ 12 mm.	d_4^{25} .	n_D^{25} .	Br (calc.).	Br (found).
Methyl	135—137°	169—171°	1.113	1.4572	27.26%	27.16%
Ethyl	144—146	175—177	1.088	1.4547	26.04	26.17
<i>iso</i> Propyl	146—148	179—181	1.056	1.4502	24.90	24.70
Propyl	149—151	184—186	1.074	1.4549	24.90	25.03
<i>iso</i> Butyl	152—154	188—190	1.051	1.4530	23.85	23.85
Butyl	154—156	193—195	1.061	1.4552	23.85	23.86
<i>iso</i> Amyl	158—160	197—199	1.050	1.4551	22.89	22.80

Δ^{α} -Dodecenoic Acid and Some of its Derivatives.—Dodecenoic acid was obtained by heating the bromo-esters with alcoholic potash in the usual manner, the yield from 100 g. of lauric acid being about 93 g. The crude acid may be used directly for the preparation of *n*-decoic acid; it may be purified by the sulphuric acid method of Fittig (*Ber.*, 1894, **27**, 2667). After redistillation under reduced pressure, it is a colourless liquid with a fruity odour, b. p. 186—189°/12 mm., m. p. 13—14°, d_4^{25} 0.905, n_D^{25} = 1.4470. It is insoluble in water but soluble in most organic solvents (Found: C, 72.05; H, 11.45. Calc. for $C_{12}H_{22}O_2$: C, 72.7; H, 11.2%). No change in physical properties was observed when the acid was further purified according to Harding and Weizmann (*J.*, 1910, **97**, 301) by recrystallisation as the barium salt from boiling alcohol, although they do not agree with the data by Zarr (*Ber. Schimmel & Co. Akt. Ges.*, 1929, 299), who prepared the same acid from decanal and malonic acid and made no attempt to purify it. The ethyl ester boils at 144—145°/15 mm.; d_4^{25} 0.8668, n_D^{25} 1.4390 (Found: C, 74.6; H, 11.7. Calc. for $C_{14}H_{26}O_2$: C, 74.3; H, 11.6%). The *amide* melts at 112.5—113.5° (Found: N, 7.1. $C_{12}H_{23}ON$ requires N, 7.2%), and the *p-toluidide* forms shining leaflets, m. p. 85—86° (Found: N, 4.9. $C_{19}H_{29}ON$ requires N, 5.2%); both of these were purified by recrystallisation from light petroleum.

n-Decoic Acid.—280 G. of dodecenoic acid were mixed in an open iron vessel with 900 g. of powdered potassium hydroxide and 50 c.c. of water, and heated with stirring until hydrogen ceased to be evolved (about 2 hours), the temperature reaching 300°. After cooling, water was added, the solution filtered, and acidified with hydrochloric acid. The layer of *n*-decoic acid was separated and distilled under reduced pressure; b. p. 161—164°/12 mm.; m. p. 30—31°; yield 170 g.