

**38. *Optically Active Alkylsuccinic Acids. Part I. Resolution of *r*-Ethylsuccinic Acid into its Optical Antipodes.***

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FOR comparison with the optically active phenyl- and diphenyl-succinic acids (J., 1916, 109, 572; 1915, 107, 406) suitable alkyl- and dialkyl-succinic acids were required. Their low specific rotation renders the known, active methyl- and dimethyl-succinic acids inconvenient for this purpose. *r*-Ethylsuccinic acid has therefore been resolved into its optical antipodes.

The inactive acid is conveniently prepared by condensing ethyl cyanoacetate with ethyl  $\alpha$ -bromobutyrate in presence of sodium ethoxide in ethyl alcohol to afford ethyl  $\alpha$ -cyano- $\beta$ -ethylsuccinate, b. p. 156°/10 mm., which is hydrolysed by boiling hydrobromic acid (48%).

*r*-Ethylsuccinic acid (26.95 g.) and quinine (59.9 g.) were dissolved in boiling alcohol (400 c.c.). The solution, on cooling, deposited 49.4 g. of crystals which were further purified by repeated crystallisation from alcohol. The course of the resolution was followed by observation of the specific rotation of the acids obtained from the successive filtrates, the following values of  $[\alpha]_D$  in acetone being obtained:  $-4.4^\circ$ ,  $-4.4^\circ$ ,  $-1.8^\circ$ ,  $+2.7^\circ$ ,  $+7.7^\circ$ ,  $+8.43^\circ$ ,  $+8.38^\circ$ . The remaining crop (13.1 g.) on decomposition with hydrochloric acid and extraction with ether gave 3.8 g. of crude *d*-ethylsuccinic acid, m. p. 69–74°,  $[\alpha]_D +19.4^\circ$ , in acetone. Repeated crystallisation of this product from benzene–light petroleum gave pure *d*-ethylsuccinic acid, m. p. 83.5–85°,  $[\alpha]_D^{18.8^\circ} +20.65^\circ$  in acetone ( $l = 2$ ,  $c = 3.7284$ ) (Found: C, 49.2; H, 6.9.  $C_6H_{10}O_4$  requires C, 49.3; H, 6.9%).

The laevorotatory by-products from the resolution described above were converted into the brucine salts, which were crystallised repeatedly from absolute alcohol. The acid obtained from the filtrates had successively  $[\alpha]_D +2.85^\circ$ ,  $+5.71^\circ$ ,  $+4.3^\circ$ ,  $-2.7^\circ$ ,  $-5.6^\circ$ ,  $-8.6^\circ$  and  $-13.4^\circ$  in acetone. The residual crop yielded a crude *l*-acid with  $[\alpha]_D -20.9^\circ$  in acetone. This, when crystallised repeatedly from benzene–light petroleum, yielded homogeneous *l*-ethylsuccinic acid, m. p. 83–85°,  $[\alpha]_D^{23.8^\circ} -20.80^\circ$  in acetone ( $l = 2$ ,  $c = 4.5944$ ) (Found: C, 49.5; H, 6.8%).

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