253. *The Resolution of Phenylmethylcarbinol.*

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dl-Phenylmethylcarbinyl hydrogen phthalate is readily resolved into the *d*- and the *l*-form by means of brucine in acetone.

THIS useful alcohol has been obtained in optically active forms by several investigators (Houssa and Kenyon, J., 1930, 2260; Ott, *Ber.*, 1928, **61**, 2139; Pickard and Kenyon, J., **1911**, **99**, 45), but as the available methods are tedious and yield only one optically active form, we re-examined the problem and eventually devised the following simple procedure.

Brucine (73 g.) was dissolved in a warm solution of *dl*-phenylmethylcarbinyl hydrogen phthalate (Houssa and Kenyon, *loc. cit.*) (50 g.) in acetone (170 c.c.) and left overnight. The crystalline material [55 g., m. p. 140° (decomp.)] which separated was recrystallised twice from the minimum amount of hot methyl acetate and obtained in needles [36 g., m. p. 153° (decomp.)]; a further 5 g. were obtained from the methyl acetate mother-liquors. This salt (41 g.), when decomposed with dilute hydrochloric acid, yielded a hydrogen phthalate (16 g.), m. p. 82°, $[\alpha]_{5461} - 74{\cdot}1°$ (*l*, 2; *c*, 5) in carbon disulphide. This was dissolved in cold carbon disulphide (30 c.c.) and left overnight in an uncorked flask; the solution was then decanted from the hard crystals of *dl*-hydrogen phthalate which had separated, warmed, and diluted with light petroleum. The optically pure *l*-phenylmethylcarbinyl hydrogen phthalate separated in clusters of fine needles (12·5 g.), m. p. 86°. Rotatory powers are in the Table.

The acetone mother-liquor from which the main portion of the brucine salt had been removed deposited, on scratching, a crop of very fine, crystalline material, m. p. 83—86° (decomp.). This (60 g.), after being twice recrystallised from acetone, was obtained in very small, irregular crystals (35 g.), m. p. 105° (decomp.); from the mother-liquors a further 10 g. were obtained. This salt (45 g.) yielded a hydrogen phthalic ester (18 g.) with $[\alpha]_{5461} + 62{\cdot}4°$ in carbon disulphide, from which by recrystallisation as described above, optically pure *d*-phenylmethylcarbinyl hydrogen phthalate (10 g.), m. p. 86° and $[\alpha]_{5461} + 79{\cdot}1°$ (*l*, 2; *c*, 2·8) in carbon disulphide, was obtained.

The combined yields of optically pure ester are about 50%.

Specific Rotatory Powers of l-*Phenylmethylcarbinyl Hydrogen Phthalate in Various Solvents at Room Temperature* (l, 2).

Solvent.	*c*.	$[\alpha]_{6438}$.	$[\alpha]_{5893}$.	$[\alpha]_{5780}$.	$[\alpha]_{5461}$.	$[\alpha]_{4358}$.
CS_2	2·835	−54·2°	−65·8°	−70·0°	−79·9°	−138·2°
,,	5·295	−52·9	−63·4	−67·5	−78·3	−134·5
C_6H_6	2·913	−12·9	−15·8	−17·20	−19·9	− 23·2
C_6H_5N	3·041	−13·1	−15·1	−15·6	−16·0	− 18·0
$CHCl_3$	3·519	—	+16·8	+19·0	+20·5	+ 49·2
EtOH	2·375	+30·2	+36·5	+40·0	+45·3	+ 96·4

These widely varying rotatory powers lie closely on a single characteristic diagram (compare Lowry's "Optical Rotatory Power," p. 418).

The *l*-hydrogen phthalic ester on hydrolysis with 5N-sodium hydroxide (2·5 mols.) yielded *l*-phenylmethylcarbinol, b. p. 93°/14 mm., in quantitative yield; its observed rotatory powers are (*l*, 0·25; *t*, 17°):

λ	6438	5893	5780	5461	4358
α_λ	−8·32°	−11·04°	−11·53°	−13·15°	−22·69°

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