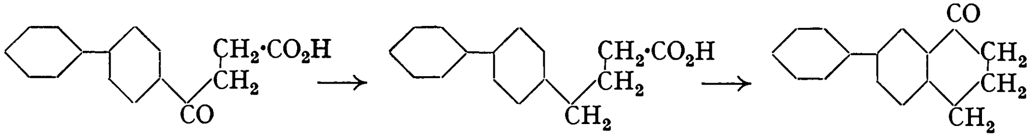
NOTE.

The Preparation of 2-Phenylnaphthalene from Diphenyl. By D. H. Hey and R. Wilkinson.

Weizmann, Bergmann, and Bograchov have recently described in a preliminary note (*Chem. and Ind.*, 1940, **59**, 402) a new method for the synthesis of 2-phenylnaphthalene. They show that diphenyl reacts with succinic anhydride to give β-p-*phenylbenzoylpropionic acid* (I) [erroneously termed β-4-diphenylylpropionic acid], which is reduced by the method of Clemmensen to yield γ-4-diphenylylbutyric acid (II) : the latter is cyclised through the acid chloride to give 1-keto-7-phenyl-1 : 2 : 3 : 4-tetrahydronaphthalene (III), which is further reduced to 7-phenyl-1 : 2 : 3 : 4-tetrahydronaphthalene and, without purification, dehydrogenated with selenium to yield 2-phenylnaphthalene.



(I.) (II.) (III.)

An almost identical series of reactions was carried out by the authors in 1936 (R. Wilkinson, Ph.D. Thesis, Manchester University, 1938) with the two-fold object of obtaining 2-phenylnaphthalene derivatives and compounds, such as 1 : 4-diketo-7-phenyl-1 : 2 : 3 : 4-tetrahydronaphthalene or 1-keto-7-phenyl-1 : 2 : 3 : 4-tetrahydronapthalene (III), which on oxidation might be expected to yield 4-phenylphthalic acid. Since both of these objectives were achieved in other ways (see Hey and Lawton, this vol., p. 374; Butterworth, Heilbron, Hey, and Wilkinson, J., 1938, 1386), further work on the condensation of succinic anhydride with diphenyl and its derivatives was temporarily abandoned. Since an early resumption of this line of work is now unlikely, it is desired to place on record the results already completed, which agree substantially with those now reported by Weizmann, Bergmann, and Bograchov (*loc. cit.*). In general the experimental conditions differ in minor detail only from those reported by Weizmann, Bergmann, and Bograchov, but whereas these authors effected the cyclisation of γ-4-diphenylylbutyric acid by conversion into the acid chloride, followed by treatment with aluminium chloride, the method now described involves the action of phosphoric oxide on the free acid in boiling benzene solution (cf. Perkin and Robinson, J., 1907, **91**, 1081; Short, Stromberg, and Wiles, J., 1936, 319).

β-p-*Phenylbenzoylpropionic Acid* (I).—An intimate mixture of diphenyl (75 g.) and succinic anhydride (50 g.) was added during ½ hour to a stirred mixture of aluminium chloride (135 g.) and nitrobenzene (500 c.c.) at 5—10°. Stirring was continued for 1 hour and after standing for 6 days at room temperature the mixture was decomposed with ice. After removal of nitrobenzene with steam the residue was extracted with aqueous sodium carbonate, and the liquid filtered. The *acid* precipitated from the filtrate on acidification was redissolved in aqueous alkali, boiled with charcoal, and reprecipitated. It crystallised from alcohol in fine needles, m. p. 185°; yield, 70% (Found : C, 75·8; H, 5·4. $C_{16}H_{14}O_3$ requires C, 75·6; H, 5·5%).

γ-4-*Diphenylylbutyric Acid* (II).—β-*p*-Phenylbenzoylpropionic acid (25 g.) in a mixture of concentrated hydrochloric acid (100 c.c.) and water (50 c.c.) was boiled for 8 hours with amalgamated zinc, prepared in the usual manner from zinc foil (100 g.). Further quantities of hydrochloric acid (50 c.c. in all) were added from time to time. The solid cake of acid was collected, washed with water, dried, and repeatedly extracted with hot light petroleum (b. p. 80—100°). The free acid separated in feathery crystals, m. p. 118—119° (Found : C, 79·9; H, 6·8. Calc. for $C_{16}H_{16}O_2$: C, 80·0; H, 6·7%).

1-*Keto*-7-*phenyl*-1 : 2 : 3 : 4-*tetrahydronaphthalene* (III).—Phosphoric oxide (25 g.) was added during ½ hour to a boiling solution of γ-4-diphenylylbutyric acid (5 g.) in benzene (75 c.c.). Boiling was continued for 3 hours, the mixture then decomposed with ice-water, and the benzene removed with steam. The oily residue was extracted with ether and, after removal of solvent, distilled at 0·01 mm. The distillate, which solidified, was washed with aqueous sodium carbonate and crystallised from light petroleum (b. p. 60—80°), from which the ketone separated in prisms, m. p. 69° (Found : C, 86·4; H, 6·3. Calc. for $C_{16}H_{14}O$: C, 86·5; H, 6·3%).—The University, Manchester. [*Received, June* 19*th*, 1940.]