NOTE.

The Condensation of Phthalic Anhydride with s.*-Octahydrophenanthrene.* By E. DE BARRY BARNETT, N. F. GOODWAY, and C. A. LAWRENCE.

o-1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-*Octahydrophenanthroylbenzoic acid* was obtained in 90% yield when 30 g. of aluminium chloride were added slowly to 19 g. of *s*.-octahydrophenanthrene and 15 g. of phthalic anhydride in 50 c.c. of tetrachloroethane. After being kept over-night and worked up in the usual way, the colourless product was recrystallised from anisole; m. p. 200° (Found : C, 79·0; H, 6·9. $C_{22}H_{22}O_3$ requires C, 79·0; H, 6·6%).

4′ : 5′-*Dichloro-octahydrophenanthroylbenzoic acid*, m. p. 276° (decomp.) after recrystallisation from anisole, was obtained in the same way from 4 : 5-dichlorophthalic anhydride (Found : C, 65·7; H, 5·0. $C_{22}H_{20}O_3Cl_2$ requires C, 65·5; H, 5·0%), but the product from 3 : 6-dichlorophthalic anhydride refused to crystallise.

When octahydrophenanthroylbenzoic acid was reduced by heating on the water-bath for 15 hours with activated zinc dust, caustic soda, and aqueous ammonia, it yielded ω-1 : 2 : 3 : 4 : 5 : 6 : 7 : 8-*octahydrophenanthryl-*o*-toluic acid*, m. p. 192° after being recrystallised from anisole (Found : C, 82·4; H, 7·7. $C_{22}H_{24}O_2$ requires C, 82·5; H, 7·5%). When this was dissolved in cold concentrated sulphuric acid (10 c.c. per g.), it was cyclised to *octahydro-*1 : 2 : 3 : 4-*dibenzanthrone*, which separated from benzene in pale yellow crystals, m. p. 200° (Found : C, 87·4; H, 7·3. $C_{22}H_{22}O$ requires C, 87·4; H, 7·3%). On heating with pyridine and acetic anhydride, this anthrone gave *octahydro-*1 : 2 : 3 : 4-*dibenzanthranyl acetate*, which crystallised from toluene in yellow needles, m. p. 214° (Found : C, 83·7; H, 7·1. $C_{24}H_{24}O_2$ requires C, 83·7; H, 7·0%).

When chromic anhydride (2·8 g.) in aqueous acetic acid was added slowly to a boiling solution of 2 g. of the above anthrone in 50 c.c. of glacial acetic acid, oxidation took place. After 3 hours' boiling, the *octahydro-*1 : 2 : 3 : 4-*dibenzanthraquinone* was collected after cooling and recrystallised from xylene, forming yellow crystals which melted, after slight sintering, to a deep red liquid at 234° (Found : C, 83·3; H, 6·5. $C_{22}H_{20}O_2$ requires C, 83·5; H, 6·3%).

The anthrone (5 g.) was reduced to *octahydro-*1 : 2 : 3 : 4-*dibenzanthracene* by boiling for 3 hours with 20 g. of activated zinc dust, 180 c.c. of alcohol, 50 c.c. of water, and 20 g. of caustic potash. After being recrystallised from glacial acetic acid (charcoal) and *cyclo*hexane, the slightly yellow crystals had m. p. 129° (Found : C, 92·1; H, 7·8. $C_{22}H_{22}$ requires C, 92·3; H, 7·7%).

Attempts to dehydrogenate octahydrodibenzanthracene with selenium and the quinone with bromine did not yield any pure product.

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