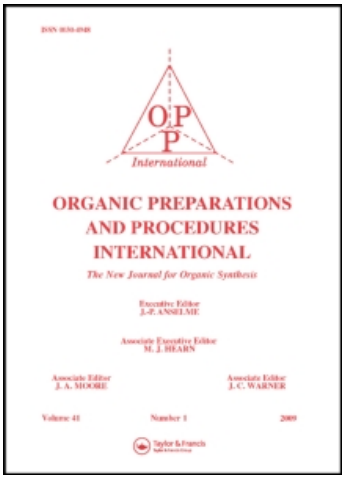

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BENZO[c]PHENANTHRENE

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217.3°, lit.[1] 217-217.4°; IR (KBr) 884, 818, 771, 740 cm^{-1}; ^1H NMR (acetone-D_6) δ 7.30-8.24 (m, 10, ArH), 8.48 (s, 2, ArH).

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BENZO[c]PHENANTHRENE

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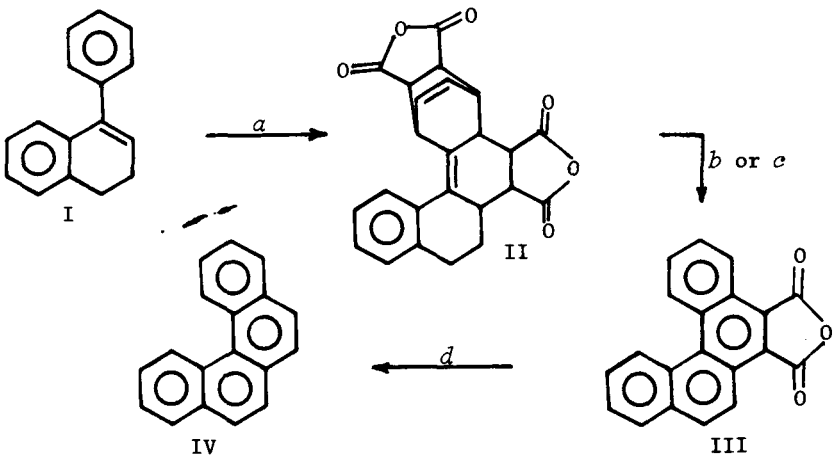
The route[1] shown was selected as being suitable for preparing several grams of benzo[c]phenanthrene (IV).[1-5] The synthesis proceeded as expected except that we experienced difficulty in removing sulfur from product III. This was overcome by substituting Pd/C (step c). The slight decrease in yield is offset by convenience and time save. We also substituted cuprous oxide and quinoline (step d, 72% yield) for barium hydroxide [lit.[1] yield

70%] with some added convenience.



[a]Maleic anhydride, Δ. [b]Pd/C, 2-methylnaphthalene, Δ. [c]S, Δ. [d]Cuprous oxide, quinoline, Δ.

EXPERIMENTAL

A solution of 1-tetralone (505 g, 3.46 mol) and 500 ml of anhydrous ether was added to an ether solution of 5.1 mol of phenyl magnesium bromide at such a rate as to maintain reflux. After addition, reflux was maintained for 1 hr. The reaction mixture was then poured onto an excess of ice cold 10% hydrochloric acid and then steam distilled to remove volatile materials. The pot residue was extracted with ether, the ether extract was concentrated and the resulting liquid was dissolved in 1 l. of benzene containing 5 g of oxalic acid. Water was removed by azeotropic distillation. The benzene solution was concentrated, passed through basic alumina, and vacuum-distilled to yield 445 g (62%) of 1-phenyl-3,4-dihydronaphthalene (I), bp. 135-140°/2 mm [lit.[6] bp. 175-177°/12 mm; 40% yield].

A 93 g (0.5 mol) sample of I and maleic anhydride (233 g, 2.38 mol) were mixed and heated to 190° for 19 hr. The resulting oil solidified and was recrystallized from acetic acid yielding 73 g (50%) of the bis Diels-

Alder adduct II, mp. 315-316° [lit.[1] mp. 315-316°; 75% yield].

The Diels-Alder adduct II (100 g, 0.34 mol) was mixed with 5 g of 5% Pd/C in 1000 ml of 2-methylnaphthalene and then heated at reflux for 8 hr. The 2-methylnaphthalene was vacuum-distilled and the resulting solid was extracted in a Soxhlet apparatus[7] with benzene to yield 74.9 g (95%) of benzo[3,4]phenanthrene-1,2-dicarboxylic acid anhydride (III), mp. 249-251° [lit.[1] 257-258; 100% yield using S].

A mixture of cuprous oxide (15.2 g, 0.11 mol), III (31.8 g, 1.1 mol), and 500 ml of quinoline was heated at reflux under nitrogen for 4 hr., cooled, and poured into 5 l. of 10% hydrochloric acid, and extracted with ether.[8] The ether solution was concentrated, dissolved in benzene, passed through basic alumina, and the benzene solution concentrated to yield 1.8 g (72%) of benzo[c]phenanthrene (IV), mp. 67.5-68° [lit.[1] mp. 68°; 70% yield].

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