

Syntheses and Reactions of Spirocyclopropaneanthrones. Part 1. Reactions of *p*-Diazo-ketones with *p*-Quinone Methides

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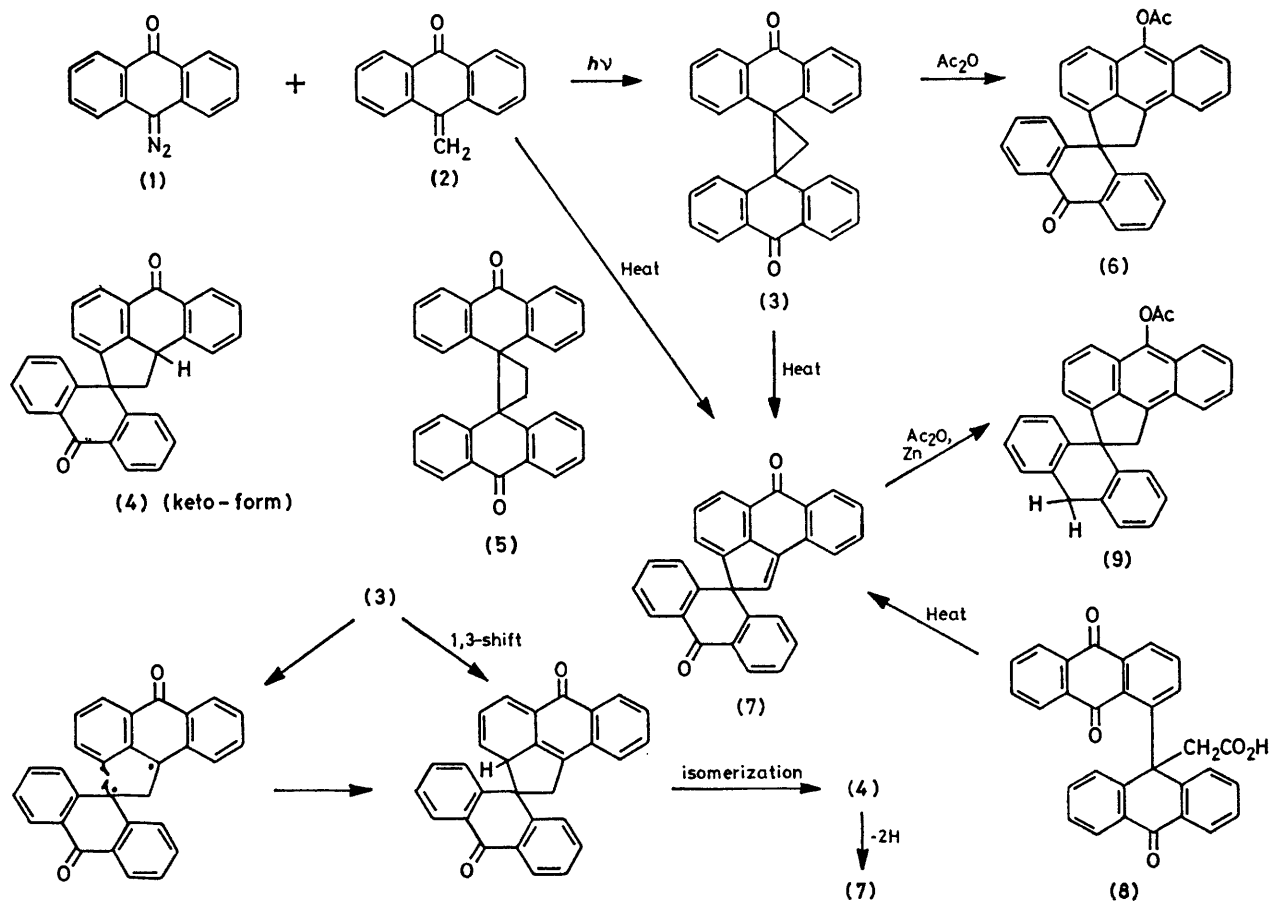
The carbenic reaction of 10-diazoanthrone (1) with 10-methyleneanthrone (2) gave dispiro[anthrone-10,1'-cyclopropane-2',10''-anthrone] (3), which thermally rearranged with synchronous ring expansion, yielding spiro[2*H*-aceanthrone-2,10'-anthrone] (7). Similar reactions of diazonaphthalen-1(4*H*)-one (11) with the quinone methide (2) and of the diazo-ketone (1) with 2,6'-di-*t*-butyl-4-methylenecyclohexa-2,5-dien-1-one (16) afforded directly the rearrangement product, 10-hydroxytribenz[*a,de,j*]anthracen-5-one (12), and the cyclopropyl ring-opening product, 10-(4-hydroxy-3,5-di-*t*-butylbenzyl)anthrone (17), respectively, instead of the dispiro-compounds.

REACTIONS of diazo-ketones and quinone methides would be expected to give dispiro-compounds containing two quinone moieties, each at the two spiro-carbons of a cyclopropane ring. This unusual system is expected to be highly reactive not only because of its high degree of strain, but also because of the possibility that aromatization of the quinone portion of the molecule might supply a strong driving force for reaction. We have therefore investigated the products from reactions of *p*-diazo-ketones with *p*-quinone methides.

RESULTS AND DISCUSSION

Irradiation of a benzene solution of 10-diazoanthrone (1) and 10-methyleneanthrone (2) under a nitrogen

atmosphere gave dispiro[anthrone-10,1'-cyclopropane-2',10''-anthrone] (3) (83%), which was slightly soluble in the usual organic solvents. In the solid state, the dispiroanthrone (3) was stable to air and prolonged heating, but it decomposed rapidly in solution. The structure of the photoreaction product was assigned as dispirocyclopropane rather than as the isomeric (4) {1,10*b*-dihydrospiro[2*H*-aceanthrone-2,10'-anthrone]} because (a) its i.r. spectrum showed the presence of appropriate carbonyl groups but no hydroxy-function, (b) it had analogous behaviour to that of dispiro[anthrone-10,1'-cyclobutane-2',10''-anthrone] (5),¹ and (c) it formed only anthraquinone on oxidation with chromium(vi) trioxide. This photoreaction involves



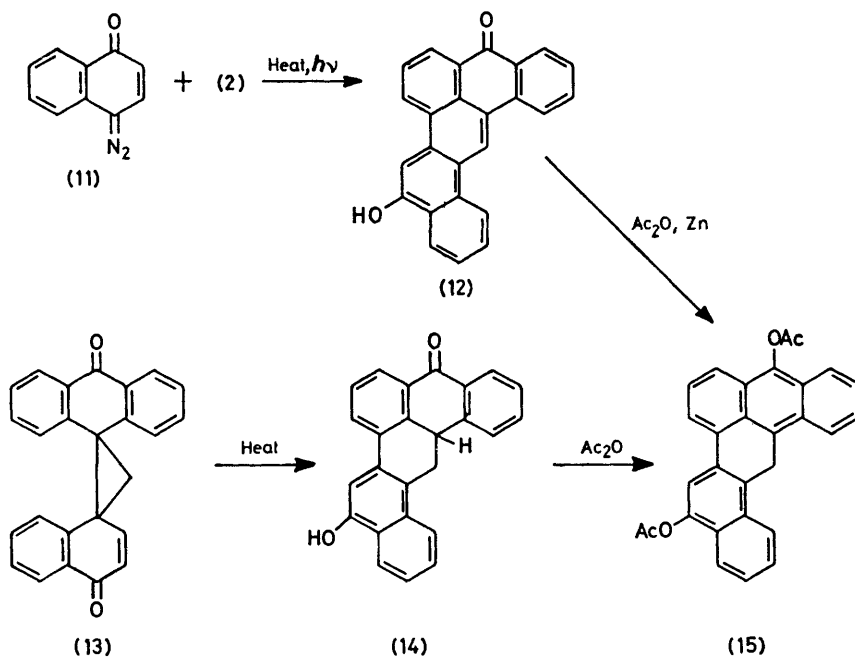
SCHEME 1

the 1,2-cycloaddition of anthronylidene carbene to the quinone methide (2), which is the sole example of a stable quinone methide having an unsubstituted methylene group.

The dispiroanthrone (3) readily rearranged in acetic anhydride with simultaneous ring expansion, yielding the acetylated product 6-acetoxyspiro[aceanthrene-2,10'-anthrone] (6) (67%). The dispiroanthrone (3) also rearranged in refluxing organic solvents, with ring expansion followed by dehydrogenation, to give spiro[2*H*-aceanthrone-2,10'-anthrone] (7) (61%). The structure of the product (7) was confirmed by direct comparison with an authentic sample prepared independently by decomposition of 10-(1-anthraquinonyl)-10-carboxymethylanthrone (8). Reductive acetylation of the

cyclization is much more favourable than the intermolecular reaction, so that dianthronylmethane (10) is not produced, and also that the cyclization proceeds so fast that the concentration of the biradical is too low to detect an e.s.r. signal. Therefore, it seems likely that the rearrangement proceeds by the biradical pathway, followed by isomerization. The intermediate (4) could not be isolated owing to its extreme reactivity, and even under a nitrogen atmosphere all attempts to isolate it led only to the spiroaceanthrone (7).

The quinone methide (2) reacted with the diazo-ketone (1) in refluxing benzene to give the spiroaceanthrone (7) (25%) along with 10,10'-bianthrone. The reaction of 10-bromo-10-bromomethylanthrone with anthrone in the presence of lithium methoxide also gave



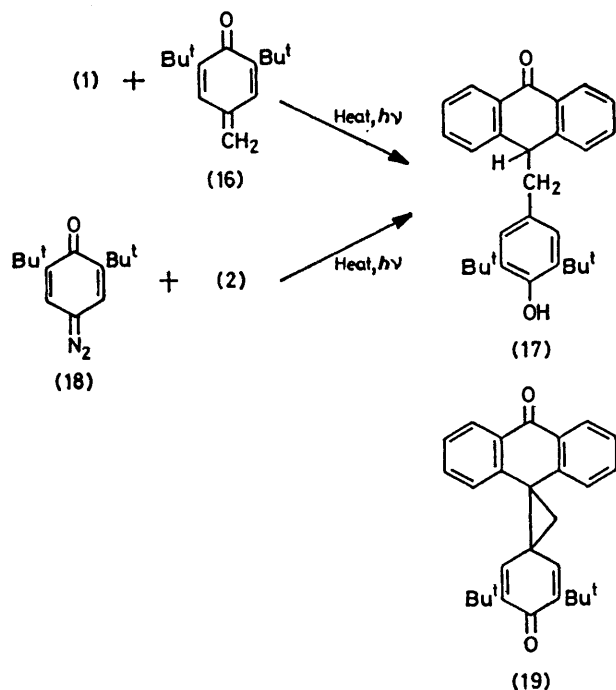
SCHEME 2

spiroaceanthrone (7) with zinc dust in acetic anhydride gave 6-acetoxyspiro[aceanthrene-3,10'-(9',10'-dihydroanthracene) (9) (51%), the carbonyl group being reduced. The thermal rearrangement of the dispiroanthrone (3) is similar to ring expansion of the spirocyclopropanefluorenes to the cyclopenta[*jk*]fluorenes² and of the dispirocyclobutaneanthrone (5) to spirobenz[*de*]anthrone.¹ For the rearrangement two pathways (the 1,3-sigmatropic and biradical pathways) are conceivable. However, it seems unlikely that the rearrangement proceeds by the former pathway, since a thermal 1,3-antarafacial shift with retention is sterically impossible. If the biradical pathway is operative, the biradical from dispiroanthrone (3) would abstract hydrogen atoms from the solvent to give dianthronylmethane (10), which was not detected in any of the thermal reaction products. In addition, e.s.r. spectroscopy failed to detect the presence of the biradical. However, it is reasonable that the intramolecular

the spiroaceanthrone (7), but in low yield. These reactions are accounted for by the initial formation of the dispiroanthrone (3), followed by rearrangement and dehydrogenation.

In the carbenic reaction of 4-diazonaphthalen-1(4*H*)-one (11) with the quinone methide (2) in benzene under a nitrogen atmosphere, the product 10-hydroxytribenz[*a,de,j*]anthracen-5-one (12), which has two crystal forms, was isolated from both photochemical and thermal reactions in 39 and 20% yields, respectively. The structure of the product (12) is assigned on the basis of its spectra, the low fluorescence emission in the visible range for both (12) and its acetate, and by analogy with the spiroaceanthrone (7) and the spirobenzanthrone¹ [from the thermal reactions of the dispiroanthrones (3) and (5), respectively]. Its structure was not pursued further. Thus, the addition of the carbene from the diazo-ketone (11) to the quinone methide (2) proceeded with rearrangement and dehydrogenation to give the tribenz-

anthrone (12) instead of the expected dispiroanthrone (13). Although (13) could not be isolated in this case, the reaction probably involves its initial formation, since the analogous dispiroanthrone (3) was isolated in the photoreaction of the diazo-ketone (1) with the quinone methide (2) and found to undergo thermal rearrangement. The rearrangement of the dispiroanthrone (13) is probably similar to that of the dispiroanthrones (3) and (5), proceeding through biradicals, followed by isomerization and dehydrogenation. The intermediates in the rearrangement could not be isolated owing to their extreme reactivity, and all attempts to isolate them led to the tribenzanthrone (12). However, acetylation of the reaction mixture in the course of the photoreaction



SCHEME 3

gave the diacetate of the intermediate (14), 5,10-diacetoxy-15*H*-tribenz[*a,de,j*]anthracene (15). The diacetate (15) could also be formed by reductive acetylation of the tribenzanthrone (12).

The carbenic reactions of the diazo-ketone (1) with 2,6-di-*t*-butyl-4-methylenecyclohexa-2,5-dien-1-one (16) afforded 10-(4-hydroxy-3,5-di-*t*-butylbenzyl)anthrone (17), together with by-products, by photochemical and thermal reactions, in 34 and 42% yields, respectively. A similar treatment of 2,6-di-*t*-butyl-4-diazocyclohexa-2,5-dien-1-one (18) with the quinone methide (2) also gave the benzylanthrone (17), but only in 3% yield. In contrast with the two foregoing dispirocyclopropyl systems [(3) and (13)], ring-opening products could be obtained, instead of the dispiro-compound and/or rearrangement products, by the above reactions. The benzylanthrone (17) was not converted into the corresponding dispiroanthrone (19) by oxidation with active manganese dioxide.³ However, it seems

reasonable that the addition of the carbenes to the quinone methides (2) or (16) involves the initial formation of the dispiroanthrone (19), rather than the biradical, as the intermediate. This view was supported by the following observations: (a) the reaction of 4-bromo-4-bromomethyl-2,6-di-*t*-butylcyclohexa-2,5-dien-1-one (20) with anthrone in the presence of lithium methoxide also gave the benzylanthrone (17); and (b) cyclopropanes were also usually produced as products in the addition of carbenes to olefins. It now appears that whether rearrangement or ring-opening of intermediate dispirocyclopropaneanthrones occurs is mainly influenced by relative hydrogen-abstracting ability of the biradicals formed from the dispiro-compounds.

EXPERIMENTAL

M.p.s were determined on a Yanagimoto hot-stage apparatus. I.r. spectra were recorded with a JASCO IRA-1 spectrophotometer (KBr disc), n.m.r. spectra with a JEOL JNM-3H-60 (60 MHz) spectrometer for solutions in deuteriochloroform (tetramethylsilane as internal standard), and mass spectra with a JEOL JMS-01SG-2 (at 75 eV). Column chromatography was carried out on Wakogel C-200 (Wako Pure Chemical Industries).

Reactions of 10-Diazoanthrone (1) with 10-Methyleneanthrone (2).—(a) *Photoreaction.* A solution of the diazo-ketone (1)⁴ (3.30 g, 15 mmol) and the quinone methide (2)⁵ (2.06 g, 10 mmol) in benzene (300 ml) was irradiated at room temperature under nitrogen with a 100-W high-pressure mercury lamp through a Pyrex filter for 4 h, during which time the approximately theoretical amount of nitrogen was evolved. The precipitate was filtered off and washed with hot benzene to remove unchanged quinone methide (2) and by-products, yielding yellow microcrystals of *dispiro*[anthrone-10,1'-cyclopropane-2',10'-anthrone] (3) (3.80 g, 83%), m.p. 139–140 °C (decomp.) (Found: C, 87.4; H, 4.75. $C_{29}H_{18}O_2$ requires C, 87.4; H, 4.55%); ν_{\max} 1665 cm^{-1} (anthrone CO); m/e 398 (M^+ , 17%), 396 (96), 206 (100), 194 (60), 178 (66), 165 (48), 78 (30), and 76 (29). The filtrate and washings were concentrated and chromatographed on silica (chloroform as eluant) to give unchanged quinone methide (2) and 10,10'-bianthrone, identical with authentic specimens.⁶

(b) *Thermal reaction.* A mixture of the diazo-ketone (1) (1.10 g, 5 mmol) and the quinone methide (2) (1.03 g, 5 mmol) in benzene (100 ml) was refluxed under nitrogen until gas evolution was complete (50 h). The mixture was chromatographed on silica (benzene and chloroform as eluant). The first eluate (benzene) contained unchanged quinone methide (2) (0.24 g, 23%). The second fraction (benzene) consisted of *spiro*[2*H*-aceanthrone-2,10'-anthrone] (7) (0.50 g, 25%), pale yellow microcrystals (from benzene), m.p. 303–304 °C (Found: C, 88.1; H, 4.3. $C_{29}H_{16}O_2$ requires C, 87.9; H, 4.1%); ν_{\max} 1643 cm^{-1} (anthrone CO); δ 6.75–8.70 (16 H, m, Ar-H and H-1); m/e 396 (M^+ , 100%), 367 (15), 339 (30), and 337 (33). The third fraction (chloroform) afforded 10,10'-bianthrone (0.20 g, 21%).

Acetylation of the Dispiroanthrone (3).—The dispiroanthrone (3) (3.98 g, 10 mmol) was suspended in acetic anhydride (100 ml) and sodium acetate (2 g) was added. The mixture was stirred and heated under reflux for 2 h, then poured into water (500 ml). The product was recrystallized from acetic acid to give 6-acetoxyspiro[ace-

anthrone-2,10'-anthrone] (6) (3.56 g, 81%), light yellow microcrystals, m.p. 288–289 °C (Found: C, 84.3; H, 4.6. $C_{31}H_{20}O_3$ requires C, 84.5; H, 4.6%); ν_{\max} 1 780 (ester CO) and 1 655 cm^{-1} (anthrone CO); δ 2.66 (3 H, s, Me), 4.44 (2 H, s, CH_2), and 6.60–8.54 (15 H, m, Ar-H); m/e 440 (M^+ , 17%), 398 (100), 339 (16), and 337 (12).

Reaction of 10-Bromo-10-bromomethylanthrone with Anthrone in the Presence of Lithium Methoxide.—To a stirred suspension of anthrone (0.20 g, 1 mmol), and lithium methoxide (0.08 g, 2 mmol) in toluene (20 ml), a solution of the dibromide⁷ (0.37 g, 1 mmol) in dioxan (50 ml) was added in small portions at room temperature. The resulting mixture was refluxed for 2 h with stirring, then poured into water (300 ml). The products were extracted (benzene), the extract was concentrated, and the residue was chromatographed on silica (benzene as eluant) to give anthraquinone (90 mg), the spiroaceanthrone (7) (6 mg, 2%), 2,3-dihydrospiro(benz[de]anthrone-3,10'-anthrone) (34 mg, 17%), m.p. 297–298 °C (lit.¹ 298–300 °C), and then 10-bromomethyl-10-methoxyanthrone as colourless microcrystals (2.10 g, 66%), m.p. 114–115 °C (Found: C, 60.7; H, 4.3; Br, 25.9. $C_{18}H_{13}BrO_2$ requires C, 60.6; H, 4.1; Br, 25.2%); ν_{\max} 1 650 cm^{-1} (anthrone CO); δ 3.00 (3 H, s, Me), 3.65 (2 H, s, CH_2), and 7.20–8.60 (8 H, m, Ar-H); m/e 318/316 (M^+ , 1), 287/285 (10), and 225/223 (100).

Reductive Acetylation of the Spiroaceanthrone (7).—A mixture of the spiroaceanthrone (7) (0.40 g, 1 mmol) and zinc dust (3 g) in acetic anhydride (20 ml) was stirred and heated under reflux for 5 h. The colour of the mixture gradually changed to yellow with a blue fluorescence. The hot mixture was filtered, and water (200 ml) was added to the filtrate. Chromatography (benzene as eluant) of the product gave yellow microcrystals of 6-acetoxyspiro[aceanthrone-2,10'-(9',10'-dihydroanthracene)] (9) (0.22 g, 52%), m.p. 265–266 °C (Found: C, 87.3; H, 5.2. $C_{31}H_{22}O_2$ requires C, 87.3; H, 5.2%); ν_{\max} 1 782 cm^{-1} (ester CO); δ 2.58 (3 H, s, Me), 3.97 (2 H, s, CH_2), 4.22 (2 H, AB quartet, H-9'), and 6.44–8.01 (15 H, m, Ar-H); m/e 426 (M^+ , 23%) and 384 (100). The reductive acetylation product (9) was obtained in 60% yield on reduction of the acetate (6) with zinc dust in acetic acid.

Thermal Reaction of the Dispiroanthrone (3).—A stirred suspension of the dispiroanthrone (3) (0.40 g, 1 mmol) in acetic acid (5 ml) was refluxed under nitrogen for 1 h. After cooling, the crystals which had separated were recrystallized (acetic acid) to give the aceanthrone (7) (0.24 g, 61%). The filtrate yielded a mixture of anthraquinone and (7), identical with authentic specimens. The intermediate (4) in this reaction could not be isolated owing to its extreme reactivity, and attempts to isolate it led to the spiroaceanthrone (7). The thermal reaction of the dispiroanthrone (3) using benzene, chlorobenzene, alcohols, chloroform, and decalin as solvents was similar to that described above.

Oxidation of the Dispiroanthrone (3) with Chromium(VI) Trioxide.—A solution of chromium trioxide (1.0 g, 10 mmol) in water (3 ml) was added to 0.40 g (1 mmol) of the dispiroanthrone (3) suspended in acetic acid (15 ml), and the resulting mixture was stirred and heated for 2 h. The hot mixture was diluted with water (150 ml) and the solid was collected to give anthraquinone (0.19 g, 46%).

Thermolysis of 10-(1-Anthraquinonyl)-10-carboxymethylanthrone (8).—The acid (8) was obtained by oxidation of dispiro[anthrone-10,1'-cyclobutane-2',10'-anthrone] (5).¹ A solution of the acid (8) (0.92 g, 2 mmol) in acetic anhydride

(30 ml) containing sodium acetate (1 g) was refluxed for 15 h. The solution was poured into water (200 ml) and the precipitate was collected and washed with water. Recrystallization from acetic acid gave the spiroaceanthrone (7) (0.51 g, 64%).

Reaction of 4-Diazonaphthalen-1(4H)-one (11) with the Quinone Methide (2).—A solution of the diazo-ketone (11)⁸ (0.85 g, 5 mmol) in benzene (100 ml) under nitrogen was heated under reflux until gas evolution was complete (48 h). The solution was concentrated to 20 ml, and after cooling, the precipitate which had formed was washed with benzene to give 10-hydroxytribenz[a,de,j]anthracen-5-one (12) as red microcrystals (0.68 g, 39%), m.p. 132–135 °C (a low-melting form) (Found: C, 86.5; H, 4.2. $C_{25}H_{14}O_2$ requires C, 86.7; H, 4.1%); ν_{\max} 3 250 (OH) and 1 630 cm^{-1} (anthrone CO); δ 6.70–8.35 (14 H, m, Ar-H, H-15, and OH); m/e 346 (M^+ , 3%), 205 (6), 193 (3), 177 (3), and 77 (100). The red microcrystals of a low-melting form were recrystallized (chloroform-hexane) to give a sample of the high-melting form of (12) as red needles, m.p. 227–229 °C. Recrystallization of the sample of high-melting form, using the low-melting form of (12) for seeding, gave red microcrystals that melted at the lower temperature.

A photoreaction of the diazo-ketone (11) (0.85 g, 5 mmol) with the quinone methide (2) (1.03 g, 5 mmol) in benzene (100 ml) also gave the tribenzanthrone (12) (0.34 g, 20%). The photolysis of the diazo-ketone (11) was complete in 6 h. In a parallel experiment, a photoreaction of the diazo-ketone (11) with the quinone methide (2) was carried out under nitrogen for ca. 2 h during which time one half of the theoretical amount of nitrogen was evolved. Acetic anhydride (30 ml) and pyridine (1 ml) were then added to the photoreaction mixture. After stirring at 90 °C for 2 h, the resulting mixture was poured into water and shaken until acetic anhydride was completely hydrolysed. The benzene solution was concentrated and chromatographed on silica (benzene as eluant). Fractional recrystallizations of appropriate fractions yielded 10-acetoxyltribenz[a,de,j]anthracen-5-one (14) (0.16 g, 8%) and 5,10-diacetoxy-10H-tribenz[a,de,j]anthracene (15) (0.08 g, 4%), identical (spectra) with the acetates from the following experiments.

Acetylation of the Tribenzanthrone (12).—The tribenzanthrone (12) (0.35 g, 1 mmol) on warming with acetic anhydride (20 ml) and pyridine (1 ml) on a steam-bath (3 h) followed by the usual work-up gave the acetate (14) as yellow microcrystals (0.21 g, 54%), m.p. 183–185 °C (Found: C, 83.3; H, 4.3. $C_{27}H_{16}O_3$ requires C, 83.5; H, 4.15%); ν_{\max} 1 768 (ester CO) and 1 646 cm^{-1} (anthrone CO); δ 2.48 (3 H, s, Me) and 6.95–8.51 (13 H, m, Ar-H and H-15); m/e 388 (M^+ , 41%), 346 (100), and 329 (19).

Reductive Acetylation of the Tribenzanthrone (12).—The procedure employed was similar to that for reductive acetylation of the spiroanthrone (7). A mixture of the tribenzanthrone (12) (0.35 g, 1 mmol) and zinc dust (3 g) in acetic anhydride (20 ml) was stirred and heated under reflux for 10 h. The product was chromatographed on silica (benzene as eluant) to give the diacetate (15) as leaflets (0.14 g, 32%), m.p. 270–272 °C (Found: C, 80.5; H, 4.8. $C_{29}H_{20}O_4$ requires C, 80.5; H, 4.7%); ν_{\max} 1 763 cm^{-1} (ester CO); δ 2.40 (3 H, s, Me), 2.65 (3 H, s, Me), 5.30 (2 H, s, CH_2), and 6.30–8.51 (12 H, m, Ar-H); m/e 432 (M^+ , 30%), 390 (100), and 348 (55).

*Reaction of the Diazo-ketone (1) with 2,6-Di-*t*-butyl-4-methylenecyclohexa-2,5-dien-1-one (16).*—(a) *Photoreaction.* The diazo-ketone (1) (2.20 g, 10 mmol) was added to a

freshly prepared solution of the quinone methide (16) (10 mmol), generated by dehydrochlorination of 2.55 g of purified 2,6-di-*t*-butyl-4-chloromethylphenol with triethylamine in benzene (400 ml),⁹ and the mixture was irradiated under nitrogen with a 100-W high-pressure mercury lamp through a Pyrex filter for 24 h at 15 °C. The solid was filtered off, and washed with hot benzene to give colourless microcrystals of 10-(4-hydroxy-3,5-di-*t*-butylbenzyl)anthrone (17) (1.42 g, 34%), m.p. 279–281 °C (decomp.) (Found: C, 84.5; H, 7.5. C₂₈H₃₂O₂ requires C, 84.4; H, 7.8%); ν_{\max} 3 620 (OH), 2 960 (t-C₄H₉), and 1 650 cm⁻¹ (anthrone CO); *m/e* 412 (M⁺, 100%), 410 (29), 219 (51), 218 (34), 203 (47), 194 (68), 175 (13), and 161 (55). Chromatography (benzene as eluant) of the filtrate gave anthraquinone (0.65 g, 31%), 10,10'-bianthrone (20 mg, 1%), 3,3',5,5'-tetra-*t*-butylstilbene-4,4'-quinone (0.29 g, 13%), and 1,2-bis-(3,5-di-*t*-butyl-4-hydroxyphenyl)ethane (22 mg, 1%), identical with authentic specimens.¹⁰

(b) *Thermal Reaction.* A solution of the diazo-ketone (1) (2.20 g, 10 mmol) and the quinone methide (16) (10 mmol) in benzene (200 ml) was heated under reflux for 24 h. The crystals were washed with hot benzene to give the benzylanthrone (17) (1.73 g, 42%).

A suspension of active manganese dioxide¹¹ (6.5 g) and the benzylanthrone (17) (0.14 g, 1 mmol) in benzene (250 ml) was shaken for 2 h. The filtrate gave unchanged (17) and anthraquinone.

*Reaction of 2,6-Di-*t*-butyl-4-diazocyclohexa-2,5-dien-1-one (18) with the Quinone Methide (2).*—A solution of the diazo-ketone (18)¹² (0.58 g, 2.5 mmol) and the quinone methide (2) (0.52 g, 2.5 mmol) in benzene (15 ml) was refluxed under nitrogen in a dark room until evolution of nitrogen ceased (10 h). After cooling, the crystals which had separated were collected to give the benzylanthrone (17) (27 mg, 3%). The filtrate was concentrated and chromatographed on silica (benzene as eluant) to give anthraquinone (0.15 g, 29%), 2,6-di-*t*-butyl-4-phenylphenol (0.22 g, 31%), 3,3',5,5'-tetra-*t*-butyl-4,4'-diphenoquinone (28 mg, 5%), and 2,6-di-*t*-butyl-1,4-benzoquinone (16 mg, 3%), identical with

authentic specimens.¹³ A photoreaction of the diazo-ketone (18) with the quinone methide (2) also gave similar result.

*Reaction of 4-Bromo-4-bromomethyl-2,6-di-*t*-butylcyclohexa-2,5-dien-1-one (20) with Anthrone in the Presence of Lithium Methoxide.*—To a stirred suspension of anthrone (0.39 g, 2 mmol) and lithium methoxide (0.15 g, 4 mmol) in toluene (20 ml), a solution of the dibromide (20)¹⁴ (0.76 g, 2 mmol) in dioxan (50 ml) was added in small portions. The resulting mixture was refluxed for 2 h, and the crystals which had separated were filtered off, washed with dioxan and then water to give the benzylanthrone (17) (0.38 g, 46%). The dibromide (20) was prepared by bromination of the quinone methide (16) in benzene with bromine.

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