PHOTOCHEMICAL REACTIONS OF PHENANTHRENEQUINONE AND DUROQUINONE WITH SUBSTITUTED 2(3*H*)-OXAZOLONES

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Received February 2nd, 1983

9,10-Phenanthrenequinone forms α-oxo-oxetane (*VIII*) when irradiated with 3-acetyl-2(3*H*)-oxazolone, and it gives adducts *X*—*XII* with 3,4,5-trisubstituted 2(3*H*)-oxazolones *IV*—*VI*. Duroquinone forms spirooxetanes *XIII* and *XIV* on photocycloaddition with *IV* and V.

Photoreactivity of quinones depends on nature of their lowest triplet states. Photochemical reactions of 1,4-quinones with alkenes produce oxetanes (usually not stable and undergoing various rearrangements[1–4]) and/or cyclobutanes[5–7]. Formation of a charge-transfer exciplex was also observed in these photocycloadditions[8]. From literature[9] it is known that 4-benzoquinone (whose lowest triplet state is n,π^*) forms predominantly oxetanes, and duroquinone (whose lowest triplet state seems to be π,π^*)[10] gives predominantly cyclobutanes. Photochemical reactions of 1,2-quinones were also investigated from mechanistic point of view[11]. Phenanthrenequinone (whose lowest triplet state is n,π^*)[12] reacts with alkenes to give α-oxo-oxetanes[13,14] (*I*) or the so called "Schönberg's adducts"[15] (*II*). These compounds are usually photolabile, and, *e.g.*, α-oxo-oxetanes rearrange on irradiation to other products depending on wavelength of the light used[16]. We carried out photochemical reactions of phenanthrenequinone and duroquinone with substituted 2(3*H*)-oxazolones *III*—*VII*. The results are given in Table I. Selective excitation of the quinone was secured by application of light with the wavelength above 370 nm (GWV filter). Phenanthrenequinone with *III*—*VI* forms products depending on substituent at the double bond in 2(3*H*)-oxazolone. If this bond is not hindered sterically (compound *III*), then [2 + 2] photocycloaddition takes place to give α-oxo-oxetane *VIII*. If the double bond is substituted with methyl (*IV*) or phenyl groups (*V*, *VI*), then [4 + 2] photocycloaddition takes place to give the Schönberg's adducts *X*—*XII*. No adduct was obtained from the photoreaction of *VII*. Dependence of formation of products on wavelength of the used light was observed in the case of compound *III*. With application of the GWV filter ($\lambda > 370$ nm) we isolated *VIII* as the only reaction product. If, however, the GWC filter ($\lambda > 300$ nm) was

used, then compound *IX* was obtained besides *VIII*. From these findings and from UV spectrum of *VIII* (the longest-wave band at 340 nm, $\log \varepsilon = 1{\cdot}7$) we conclude that *IX* is formed by subsequent reactions of the primary photolysis product *VIII*. The photolysis of α-oxo-oxetanes itself is a complex reaction depending on arrangement of the experiment[16]. It takes place on direct irradiation and can also be sensibilized by the reaction products and phenanthrenequinone itself. In our case the reaction mixture contains compound *III* which, being an acetylating agent[32], could acetylate hydroxyl group of the primary formed photolysis product. Yields of the adducts, *i.e.* *X* < *XI* < *XII*, agree with the reaction going through the most stable diradical. In no case of the photochemical reactions of duroquinone we could observe formation of cyclobutane ring, which could be expected with respect to π, π^* nature of its lowest triplet state. Compound III gave a polymer, compounds *IV*, *V* gave the corresponding spirooxetanes *XIII*, *XIV*, no experiments were carried out with compound *VI*, and compound *VII* gave no photocycloaddition product. Formation of spirooxetanes in photoreactions of duroquinone was observed earlier[17,18], and it is explained by rearrangement of the photochemically labile 2,5-cyclohexadienone grouping[19] formed after photocycloadditon of carbonyl groups to alkene (in our case to the respective 2(3*H*)-oxazolones *IV* and *V*). Such photorearrangements of 2,5--cyclohexadienones are well investigated also from mechanistic point of view[20]. Structure of the products was suggested on the basis of spectral data. IR spectrum of compound *VIII* contains vibrations of carbonyl group of oxazolidone ring at 1 808 cm^{-1} and a combined band at 1 715 cm^{-1} belonging to acetyl group and carbonyl group of the phenanthrenequinone residue. The presence of 3-acetyl-2(3*H*)-oxazolone ring in *IX* is indicated by the bands of its carbonyl groups at 1 760 and 1 720

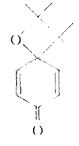
TABLE I

Products of photoreactions of 2(3*H*)-oxazolones *III* – *VII* with phenanthrenequinone and duroquinone

Quinone E_T, kJ mol^{-1}	*III*	*IV*	*V*	*VI*	*VII*
Phenanthrenequinone 272[a], 201[b]	*VIII* 60% *IX* 12%	*X* 37%	*XI* 77%	*XII* 98%	0[c]
Duroquinone 216[d]	polymers	*XIII* 6%	*XIV* 30%	[e]	0

[a] The value obtained from quenching experiments[23]; [b] the value obtained from phosphorescence spectrum[24]; [c] no cycloaddition reaction was observed; [d] the value taken from ref.[17]; [e] the reaction was not carried out.

cm^{-1}. The given structure also agrees with the fact that the spectrum lacks vibrations of carbonyl group of phenanthrenequinone residue. The spirooxetanes *XIII* and *XIV* have, in their IR spectra, four characteristic bands in the region 1 600 – 1 800 cm^{-1}. The first two are assigned to the 2-oxazolidone section of the molecule (1 780 cm^{-1} the ring carbonyl, 1 720 cm^{-1} acetyl), and the other two bands are assigned (on the basis of the data[20]) to the cyclopentene grouping (1 700 cm^{-1} carbonyl, 1 620 cm^{-1} C=C). The IR spectra of the Schönberg's adducts contain, besides the vibrations of carbonyl groups, further two characteristic bands (about 1 660 cm^{-1} and about 1 610 cm^{-1}) which are assigned to skeletal vibrations of C=C bonds of the phenanthrene section of the molecule[15]. UV spectra of *X – XII* have vibrational structure characteristic for this type of compounds[15]. Compound *IX* has similar spectrum, its structure being in this respect actually identical. Enone grouping of compounds-



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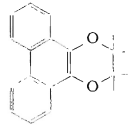
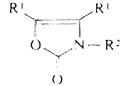
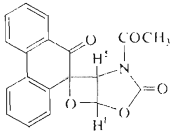


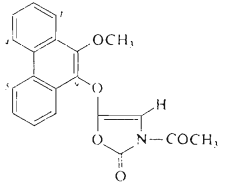
II



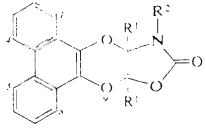
III, R^1 = H, R^2 = CH_3CO
IV, R^1 = CH_3, R^2 = CH_3CO
V, R^1 = C_6H_5, R^2 = CH_3CO
VI, R^1 = R^2 = C_6H_5
VII, R^1 = H, R^2 = C_6H_5



VIII

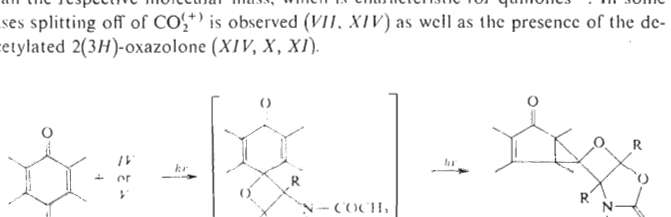


IX



X, R^1 = CH_3, R^2 = CH_3CO
XI, R^1 = C_6H_5, R^2 = CH_3CO
XII, R^1 = R^2 = C_6H_5

XIII and *XIV* is manifested by absorption about 300 nm in accordance with literature data for similar structures[17]. The ^{1}H NMR spectra of photoadducts of phenanthrenequinone with 2(3*H*)-oxazolones contain multiplet signals of aromatic protons of the phenanthrene residue. The signal at 8·6δ indicates the presence of double bond between $C_{(9)}$ and $C_{(10)}$ atoms of phenanthrene and belongs to H^4 and H^5 protons. If this signal is absent from the spectrum,then the system is obviously non-planar (it is not phenanthrodioxene[15]). The protons signals due to H^1 and H^8 in the ^{1}H NMR spectrum of *IX* are resolved, since the protons have different environment. The same is true of the spectrum of *XII*, which is probably due to phenyl group at nitrogen (in the case of *XI* the H^1 and H^8 signals form a common multiplet). Two doublets of the H^1 and H^5 protons of oxazolidone section of molecule *VIII* differ by half-width of the individual lines of the doublet. The H^5 doublet has a half-width about 4 Hz, whereas that of H^1 has 2 Hz. This fact can be due to interaction of H^5 with the phenanthrene section of the molecule[21]. All the adducts except for *IX* have the molecular ions in their mass spectra. In the case of *IX* a fragment with $m/z = 321$ can only be observed which is due to splitting off of CO. Generally it is possible to observe splitting off of a ketene molecule($M^{+\cdot} - 42$),splitting off of $CH_3CO^{(+)}$ ($m/z = 43$), and decomposition of the adducts to the original components. The quinone fragment is observed at a m/z value higher by two units than the respective molecular mass, which is characteristic for quinones[22]. In some cases splitting off of $CO_2^{(+)}$ is observed (*VII*, *XIV*) as well as the presence of the deacetylated 2(3*H*)-oxazolone (*XIV*, *X*, *XI*).



XIII, R = CH_3
XIV, R = C_6H_5

EXPERIMENTAL

The starting 2(3*H*)-oxazolones (*III*) (ref.[25]), (*IV*) (ref.[26]), (*V*) (ref.[27]), (*VI*) (ref.[27]), (*VII*) (ref.[28]), 9,10-phenanthrenequinone[29], and duroquinone[30] were prepared according to the literature data. The photochemical preparations were carried out in a 150 ml reactor[31] with GWV and GWC filters ($\lambda > 370$ nm and $\lambda > 330$ nm, respectively). Before, the reaction, the mixture was treated with nitrogen gas (15 min) and then irradiated with a medium-pressure Hg discharge lamp Tesla RVK 125 at 20°C. The reaction course was followed by TLC (Silufol UF_{254}). The melting points were determined with a Kofler apparatus and are not corrected. The IR spectra were measured wit a UR-20 apparatus (Zeiss, Jena), the UV spectra were measured with a UV-VIS

Specord (the ε quantity is given in $m^2\ mol^{-1}$), the ^1H NMR spectra, (δ, ppm) were measured with a Tesla BS 487 C apparatus (80 MHz). The chemical shifts are given with respect to tetramethylsilane as standard. The mass spectra (70 eV, 100 μA) were measured with an MS 902 S apparatus (AEI, Manchester).

10-Oxo-9,10-dihydrophenanthrene-⟨9-spiro-6⟩-3-oxo-4-acetyl-2,7-dioxa-4-azabicyclo-[3,2,0]heptane (*VIII*) and 3-acetyl-5-(10-methoxy-9-phenanthryloxy)-2(3*H*)-oxazolone (*IX*)

0·64 g ($5 . 10^{-3}$ mol) *III* and 1·04 g ($5 . 10^{-3}$ mol) phenanthrenequinone in 150 ml benzene were irradiated through a GWC filter until discolouration of the solution (about 1 h). The solvent was evaporated, and the residue was recrystallized twice from acetonitrile to give 1 g (60%) *VIII*, m.p. 233°C (decomp.). IR spectrum (chloroform): 1 808, 1 715, 1 605, 1 370, 1 300, 1 140 cm^{-1}. UV spectrum (methanol): λ_{max} 208 nm (log ε 3·36), 248 (3·47), 253 (3·47), 272 (2·74), 283 (2·70), 294 (2·48), 310 (2·00), 340 (1·70). ^1H NMR spectrum (deuteriochloroform): 2·10 (s, 3 H, CH_3), 5·15 (d, 1 H, H^5), 6·51 (d, 1 H, H^1), 7·40—8·20 (m, 8 H, arom.), $J_{1,5}$ = 4·2 Hz. Mass spectrum, *m/z* (rel. int.): 335 (23), 263 (15), 222 (14), 221 (37), 220 (46), 210 (21), 209 (100), 181 (15), 180 (39), 165 (21), 163 (10), 152 (17), 151 (12), 149 (33), 88 (10), 73 (12), 71 (12), 60 (14), 57 (21), 55 (15), 44 (63), 43 (73), 41 (21), 39 (10), 28 (48). For $C_{19}H_{13}NO_5$ (335·3) calculated: 68·06% C, 3·91% H, 4·18% N; found: 68·17% C, 4·08% H, 4·37% N. The residue was submitted to column chromatography (silica gel, chloroform) and gave, as the main fraction, 0·4 g (12%) *IX*, m.p. 170—171°C (cyclohexane). IRspectrum (chloroform): 1 760, 1 720, 1 655, 1 610, 1 415, 1 370, 1 150, 1 080 cm^{-1}. UV spectrum (methanol): λ_{max} 211 nm (log ε 3·49), 226 (3·52), 251 (3·67), 257 (3·73), 274 (3·10), 280 (2·98), 293 (2·93), 305 (2·93). ^1H NMR spectrum (deuteriochloroform): 2·73 (s, 3 H, CH_3), 3·48 (s, 3 H, CH_3O), 7·33 (s, 1 H, CH of oxazolone), 7·60—7·85 (m, 4 H, arom.), 8·00—8·20 (m, 1 H, arom). 8·35—8·50 (m, 1 H, arom.), 8·58—8·75 (m, 2 H, arom.). Mass spectrum, *m/z* (rel. int.): 322 (10), 321 (41), 249 (10), 248 (44), 237 (21), 236 (100), 228 (12), 221 (82), 207 (14), 165 (29). 164 (15), 163 (12), 57 (18), 55 (12), 43 (29), 41 (15), 28 (21). For $C_{20}H_{15}NO_5$ (349·3) calculated: 68·76% C, 4·33% H, 4·01% N; found: 68·40% C, 4·71% H, 4·30% N.

11-Oxo-12-acetyl-9a,12a-dimethyl-9a,11,12,12a-tetrahydrooxazo[4,5-*b*]phenanthro[9,10-*e*]-1,4-dioxine (*X*)

1·0 g ($6·5 . 10^{-3}$ mol) *IV* and 1·4 g ($6·5 . 10^{-3}$ mol) phenanthrenequinone in 100 ml benzene were irradiated through the GWV filter for 18 h. The solvent was evaporated, and the residue was crystallized twice from dibutyl ether to give 0·9 g (37%) *X*, m.p. 210°C. IR spectrum (chloroform): 3 095, 1 798, 1 730, 1 658, 1 610, 1 455, 1 325, 1 125 cm^{-1}. UV spectrum (methanol): λ_{max} 212 nm (log ε 3·82), 222 (3·66), 250 (3·68), 257 (3·78), 273 (3·20), 284 (2·90), 295 (2·96), 309 (3·04). ^1H NMR spectrum (deuteriochloroform): 2·05 (s, 3 H, CH_3), 2·26 (s, 3 H, CH_3), 2·29 (s, 3 H, CH_3), 7·55—7·70 (m, 4 H, arom). 8·10—8·25 (m, 2 H, arom.), 8·55—8·75 (m, 2 H, arom.). Mass spectrum, *m/z* (rel. int.): 363 (16), 210 (21), 209 (63), 181 (13), 152 (13), 149 (11), 113 (29, 44 (12), 41 (11), 32 (12), 28 (100). For $C_{21}H_{17}NO_5$ (363·4) calculated: 69·41% C, 4·72% H, 3·86% N; found: 69·29% C, 4·75% H, 3·90% N.

11-Oxo-12-acetyl-9a,12a-diphenyl-9a,11,12,12a-tetrahydrooxazo[4,5-*b*]phenanthro[9,10-*e*]-1,4-dioxine (*XI*)

0·84 g ($3 . 10^{-3}$ mol) *V* and 0·6 g ($3 . 10^{-3}$ mol) phenanthrenequinone in 100 ml benzene were irradiated through the GWV filter for 5 h (until discolouration of the solution). The solvent was evaporated and the residue was recrystallized from dioxane to give 1·1 g (77%) *XI*, m.p. 255 to 256°C. IR spectrum (chloroform): 1 800, 1 730, 1 660, 1 608, 1 460, 1 375, 1 340, 1 306, 1 160,

1 125 cm^{-1}. UV spectrum (methanol): λ_{max} 223 nm (log ε 4·43), 250 (3·56), 257 (4·64), 270 (3·18), 295 (2·92), 296 (3·01), 310 (3·09), 344 (2·00). ^1H NMR spectrum (deuteriochloroform): 2·35 (s, 3 H, CH_3), 7·05–7·42 (m, 10 H, 2 × C_6H_5), 7·50–7·80 (m, 4 H, arom.), 8·10–8·40 (m, 2 H, arom.), 8·60–8·80 (m, 2 H, arom.). Mass spectrum, *m/z* (rel. int.): 487 (12), 238 (17), 237 (100), 209 (12), 181 (12), 180 (13), 152 (12), 105 (35), 104 (38), 77 (30), 43 (27), 28 (65). For $C_{31}H_{21}NO_5$ (487·5) calculated: 76·38% C, 4·34% H, 2·87% N; found: 75·36% C, 4·55% H, 3·07% N.

11-Oxo-9a,12,12a-triphenyl-9a,11,12,12a-tetrahydrooxazo[4,5-*b*]phenanthro[9,10-*e*]-1,4-dioxine (*XII*)

1·0 g (3 . 10^{-3} mol) *VI* and 0·63 g (3 . 10^{-3} mol) phenanthrenequinone in 100 ml benzene were irradiated through the GWC filter for 9 h. The solvent was evaporated, and the residue was submitted to column chromatography (alumina, act. II, benzene) to give 1·6 g (98%) *XII*, m.p. 233–234°C (benzene–ethanol 1 : 1). IR spectrum (chloroform): 1 785, 1 660, 1 515, 1 460, 1 360, 1 340, 1 120, 1 120, 1 608, 1 355 cm^{-1}. UV spectrum (methanol): λ_{max} 218 nm (log ε 3·70), 248 (3·60), 258 (3·72), 270 (3·50), 283 (3·15), 297 (3·26), 309 (3·30). ^1H NMR spectrum (deuteriochloroform): 7·00–7·50 (m, 15 H, 3 × C_5H_4), 7·50–7·75 (m, 4 H, arom.), 7·80–7·95 (m, 1 H, H^1), 8·20–8·40 (m, 1 H, H^8), 8·55–8·75 (m, 2 H, H^4 and H^5). Mass spectrum, *m/z* (rel. int.): 521 (2), 314 (14), 313 (69), 180 (61), 165 (14), 78 (100), 77 (61), 57 (17), 51 (30), 50 (22), 45 (16), 43 (16), 41 (14), 39 (19), 31 (33), 28 (22). For $C_{25}H_{23}NO_4$ (521·6) calculated: 80·60% C, 4·44% H, 2·89% N; found: 80·60% C, 4·61% H, 2·72% N.

1,3,4,5-Tetramethylbicyclo[3,1,0]-3-hexen-2-one-⟨6-spiro-6⟩-4-acetyl-1,5-dimethyl-2,7-dioxa-4-azabicyclo[3,2,0]heptan-3-one (*XIII*)

1·55 g (0·01 mol) *IV* and 1·64 g (0·01 mol) duroquinone in 100 ml acetone were irradiated through the GWV filter for 140 h. The solvent was evaporated, and the residue was submitted to column chromatography (silica gel, benzene) to give 0·18 g (6%) *XIII*, m.p. 204–205°C (diethyl ether). IR spectrum (KBr): 2 900, 1 780, 1 720, 1 680, 1 640, 1 380, 1 370, 1 310, 1 290, 1 130, 1 075, 960 cm^{-1}). UV spectrum (methanol): λ_{max} 205 nm (log ε 2·90), 248 (2·98), 285 (2·30). ^1H NMR spectrum (deuteriochloroform): 1·25, 1·42, 1·71, 1·75, 1·92, 2·10 (s, 3 H, 6 × CH_3), 2·50 (s, 3 H, CH_3CO). Mass spectrum, *m/z* (rel. int.): 319 (15), 208 (26), 167 (11), 166 (100), 165 (79), 164 (13), 147 (13), 137 (11), 136 (11), 114(11), 113 (11), 112 (11), 44(10), 43 (34), 42 (12), 28 (21). For $C_{17}H_{21}NO_5$ (319·4) calculated: 63·93% C, 6·63% H, 4·39% N; found: 64·00% C, 6·60% H, 4·42% N.

1,3,4,5-Tetramethylbicyclo[3,1,0]-3-hexen-2-one-⟨6-spiro-6⟩-4-acetyl-1,5-diphenyl-2,7-dioxa-4-azabicyclo[3,2,0]heptan-3-one (*XIV*)

0·84 g (3 . 10^{-3} mol) *V* and 0·5 g (3 . 10^{-3} mol) duroquinone in 100 ml acetone were irradiated through the GWV filter for 24 h. The solvent was evaporated, and the residue was submitted to column chromatography (silica gel, benzene–ethanol 10 : 1) to give 0·4 g (30%) *XIV*, m.p. 228–230°C (ethanol). IR spectrum (chloroform): 1 805, 1 730, 1 700, 1 640, 1 310, 1 285 cm^{-1}. UV spectrum (methanol): λ_{max} 208 nm (log ε 3·39), 240 (2·83), 263 (2·56), 330 (1·30). ^1H NMR spectrum (deuteriochloroform): 1·10 (s, 3 H, CH_3), 1·38–1·41 (m, 9 H, 3 × CH_3), 2·55 (s, 3 H, CH_3CO), 7·10–7·30 (m, 10 H, 2 × C_6H_5). Mass spectrum, *m/z* (rel. int.): 443 (0·1), 402 (17), 238 (20), 237 (100), 136 (53), 121 (11), 105 (30), 104 (30), 93 (14), 77 (33), 44 (33), 28 (20). For $C_{27}H_{25}NO_5$ (443·5) calculated: 73·12% C, 5·68% H, 3·16% N; found: 72·35% C, 6·64% H, 3·43% N.

This study was partially conducted by one of the authors (J. K.) at the Max Planck Institute für Kohlenforschung, Abt. Strahlenchemie, Mülheim/Ruhr, BRD. We are grateful to the Max Planck Society for fellowship.

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Translated by J. Panchartek.