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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1252). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Photoproducts Derived from 11,12-Dibenzoyl-9,10-dihydro-9,10-dimethoxy-9,10-ethenoanthracene

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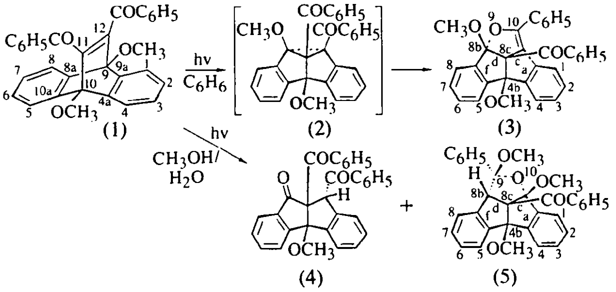
Abstract

Photolysis of 11,12-dibenzoyl-9,10-dihydro-9,10-dimethoxy-9,10-ethenoanthracene, (1), in a mixture of benzene and methanol gives the dibenzofuranopentalene (3), whereas irradiation of (1) in a mixture of benzene and aqueous methanol gives a mixture of the dibenzopentalenone (4) and the dibenzofuranopentalene derivative (5). The structures of two of these photoproducts, namely, the methanol solvate of 8c-benzoyl-8b,8c-dihydro-4b,8b-dimethoxy-4b*H*-dibenzo[*a*,*f*]furano[3,4,5-*c*,*d*]pentalene, $C_{32}H_{24}O_4.0.5CH_3OH$, (3), and 8c-benzoyl-8b,8c,8d,9-tetrahydro-8b,8d,9-trimethoxy-4b*H*-dibenzo[*a*,*f*]furano[3,4,5-*c*,*d*]pentalene, $C_{33}H_{28}O_5$, (5), have been determined by X-ray diffraction methods.

Comment

It has been reported previously that the photolysis of the dibenzobarrelene (1) in benzene, methanol or acetone leads to the dibenzosemibullvalene derivative (2) (Murty, Pratapan, Kumar, Das & George, 1985). In a recent study, it was shown that the photolysis of (1) gives rise to different products depending on the reaction conditions (Kumar, Asokan, Mathew, Ramaiah, Das, Rath & George, 1996). Thus, photolysis of (1) in a mixture of methanol and benzene (4:1) gives a 90% yield of (3) whereas irradiation of (1) in aqueous methanol gives a mixture of (4) (40%) and (5) (34%).



The present study was undertaken in order to confirm the structures of compounds (3) and (5). Compound (3) may arise through the opening of the cyclopropane ring in (2) followed by ring closure analogous to the phototransformations of 9-aryl-substituted dibenzobarrelenes (Pratapan, Ashok, Gopidas, Rath, Das & George, 1990). Both (4) and (5) may arise through an ionic cleavage of the cyclopropane ring in (2) followed by the loss of a methyl group in the case of (4) and the addition of methanol in the case of (5).

Both (3) and (5) contain a dibenzopentalenofuran skeleton, incorporating a pentacyclic fused-ring core containing two six- and three five-membered rings, which has the shape of a shallow bowl in each case (Figs. 1 and 2). In the case of (3), the methoxy groups at the C4b and C8b positions and the benzoyl group at the C8c position lie on the outer side of the bowl whereas in (5), the benzoyl group at C8c and the methoxy substituent at C4b are *cis* with respect to one another. The 'butterfly angles' formed by the fusion of the two indane systems are 106.5 and 112.3° for compounds (3) and (5), respectively.

Compound (3) crystallizes as a hemisolvate with half a molecule of methanol per molecule. The solvent molecule exhibits large anisotropic thermal motion owing to disorder which could not be resolved. The occupancy factor was refined for the solvent molecule.

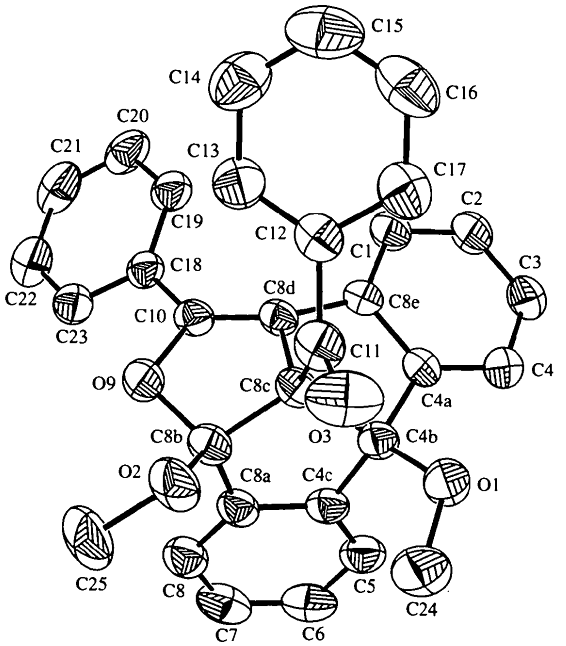


Fig. 1. View of (3) showing the labeling of the non-H atoms. Displacement ellipsoids are shown at 40% probability levels. For clarity, the solvent of crystallization has not been shown.

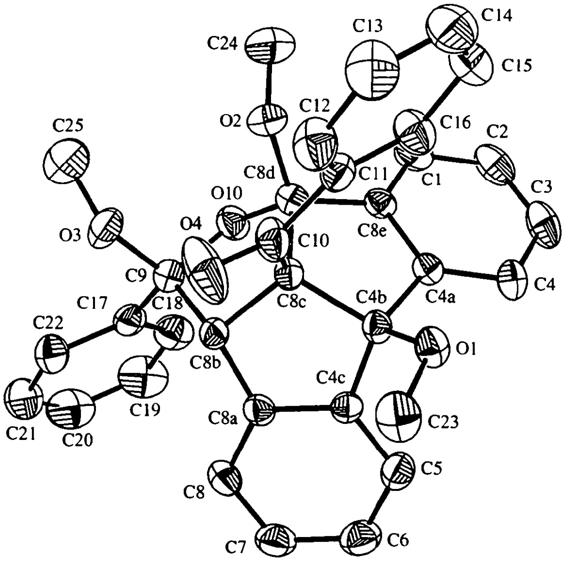


Fig. 2. View of (5) showing the labeling of the non-H atoms. Displacement ellipsoids are shown at 40% probability levels.

Experimental

Compounds (3) and (5) were prepared according to Kumar *et al.* (1996).

Compound (3)

Crystal data

$C_{32}H_{24}O_4.0.5CH_4O$
$M_r = 488.53$
Monoclinic
$P2_1/n$
$a = 11.664\ (4)$ Å
$b = 17.476\ (6)$ Å
$c = 12.408\ (4)$ Å
$\beta = 94.81\ (3)°$
$V = 2520.3\ (15)$ Å^3
$Z = 4$
$D_x = 1.287$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ (graphite-monochromated) radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.0–12.5°$
$\mu = 0.085$ mm^{-1}
$T = 293\ (2)$ K
Irregular
$0.40 \times 0.30 \times 0.30$ mm
Colorless

Data collection

Siemens $R3$ diffractometer
$\omega–2\theta$ scans
Absorption correction: none
6062 measured reflections
5786 independent reflections
1843 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0387$

$\theta_{\rm max} = 27.50°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 24$
$l = -17 \rightarrow 17$
3 standard reflections monitored every 100 reflections
intensity decay: 4%

Refinement

Refinement on F^2
$R(F) = 0.0435$
$wR(F^2) = 0.0816$
$S = 0.777$
5784 reflections
338 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.01P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.023$
$\Delta\rho_{\rm max} = 0.202$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.180$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (3)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.35210 (14)	0.64422 (9)	0.40267 (13)	0.0638 (5)
O2	0.20556 (15)	0.76449 (10)	0.15820 (13)	0.0707 (6)
O3	0.1834 (2)	0.60704 (11)	0.2336 (2)	0.0908 (7)
O9	0.08777 (13)	0.83452 (9)	0.26311 (12)	0.0565 (5)
C1	0.0575 (2)	0.72702 (13)	0.6134 (2)	0.0532 (7)
C2	0.1086 (2)	0.69257 (14)	0.7067 (2)	0.0614 (8)
C3	0.2173 (2)	0.66227 (15)	0.7090 (2)	0.0616 (8)
C4	0.2801 (2)	0.66550 (14)	0.6189 (2)	0.0555 (7)
C4a	0.2293 (2)	0.69870 (13)	0.5262 (2)	0.0440 (6)
C4b	0.2820 (2)	0.70882 (14)	0.4186 (2)	0.0471 (7)
C4c	0.3425 (2)	0.78508 (15)	0.4078 (2)	0.0487 (7)
C5	0.4344 (2)	0.8119 (2)	0.4751 (2)	0.0609 (8)
C6	0.4759 (2)	0.8847 (2)	0.4568 (2)	0.0719 (9)
C7	0.4245 (3)	0.9297 (2)	0.3742 (2)	0.0752 (9)
C8	0.3324 (2)	0.9032 (2)	0.3082 (2)	0.0614 (8)
C8a	0.2926 (2)	0.82890 (15)	0.3235 (2)	0.0492 (7)
C8b	0.1925 (2)	0.78832 (15)	0.2639 (2)	0.0523 (7)
C8c	0.1712 (2)	0.72095 (13)	0.3385 (2)	0.0450 (6)
C8d	0.0909 (2)	0.76042 (14)	0.4124 (2)	0.0425 (6)
C8e	0.1182 (2)	0.72981 (13)	0.5218 (2)	0.0421 (6)
C10	0.0469 (2)	0.82336 (15)	0.3647 (2)	0.0453 (6)
C11	0.1242 (2)	0.6460 (2)	0.2875 (2)	0.0590 (8)
C12	0.0074 (2)	0.6190 (2)	0.3098 (2)	0.0536 (7)
C13	−0.0902 (3)	0.6614 (2)	0.2838 (2)	0.0673 (8)
C14	−0.1971 (3)	0.6324 (2)	0.3018 (3)	0.0891 (11)
C15	−0.2053 (3)	0.5615 (2)	0.3489 (3)	0.0990 (12)
C16	−0.1090 (3)	0.5194 (2)	0.3754 (2)	0.0911 (11)
C17	−0.0023 (3)	0.5474 (2)	0.3551 (2)	0.0703 (8)
C18	−0.0332 (2)	0.88102 (15)	0.3953 (2)	0.0460 (7)

C19	−0.1140 (2)	0.8654 (2)	0.4684 (2)	0.0584 (8)
C20	−0.1886 (2)	0.9217 (2)	0.4981 (2)	0.0734 (9)
C21	−0.1845 (2)	0.9941 (2)	0.4550 (2)	0.0795 (10)
C22	−0.1058 (2)	1.0102 (2)	0.3821 (2)	0.0722 (9)
C23	−0.0303 (2)	0.9546 (2)	0.3530 (2)	0.0577 (7)
C24	0.4231 (2)	0.64872 (15)	0.3135 (2)	0.0840 (10)
C25	0.2200 (2)	0.8231 (2)	0.0811 (2)	0.0984 (11)
C1*S*†	0.0066 (13)	−0.0143 (10)	0.004 (2)	0.220 (4)
O1*S*†	0.1234 (6)	−0.0035 (4)	0.0535 (7)	0.220 (4)

† Occupancy = 0.480 (3).

Table 2. *Selected geometric parameters (Å, °) for (3)*

O1—C4b	1.418 (2)	C4b—C8c	1.577 (3)
O1—C24	1.439 (2)	C4c—C8a	1.385 (3)
O2—C8b	1.397 (2)	C8a—C8b	1.506 (3)
O2—C25	1.422 (3)	C8b—C8c	1.531 (3)
O3—C11	1.211 (3)	C8c—C8d	1.529 (3)
O9 C10	1.398 (2)	C8c—C11	1.534 (3)
O9—C8b	1.463 (3)	C8d—C10	1.331 (3)
C4a—C8e	1.402 (3)	C8d—C8e	1.470 (3)
C4a—C4b	1.526 (3)	C10—C18	1.446 (3)
C4b—C4c	1.519 (3)	C11—C12	1.490 (3)
C4b—O1—C24	116.1 (2)	C8b—C8c—C11	118.5 (2)
C8b—O2—C25	116.5 (2)	C8d—C8c—C4b	101.2 (2)
C10—O9—C8b	105.3 (2)	C8b—C8c—C4b	108.6 (2)
C8e—C4a—C4b	111.1 (2)	C11—C8c—C4b	113.0 (2)
O1—C4b—C4c	114.1 (2)	C10—C8d—C8e	139.2 (2)
C4c—C4b—C4a	113.7 (2)	C10—C8d—C8c	109.8 (2)
O1—C4b—C8c	118.1 (2)	C8e—C8d—C8c	107.1 (2)
C4c—C4b—C8c	100.9 (2)	C1—C8e—C4a	119.2 (2)
C8a—C4c—C4b	112.5 (2)	C1—C8e—C8d	133.1 (2)
O2—C8b—O9	108.4 (2)	C4a—C8e—C8d	107.8 (2)
O2—C8b—C8a	117.4 (2)	C8d—C10—O9	111.6 (2)
O9—C8b—C8a	110.8 (2)	C8d—C10—C18	133.8 (2)
O2—C8b—C8c	112.0 (2)	O9—C10—C18	114.6 (2)
O9—C8b—C8c	104.5 (2)	O3—C11—C12	119.8 (3)
C8a—C8b—C8c	102.9 (2)	O3—C11—C8c	120.5 (3)
C8d—C8c—C8b	98.9 (2)	C12—C11—C8c	119.6 (2)
C8d—C8c—C11	114.6 (2)		

Compound (5)

Crystal data

$C_{33}H_{28}O_5$
$M_r = 504.55$
Orthorhombic
Pbca
$a = 17.513$ (5) Å
$b = 14.897$ (6) Å
$c = 19.587$ (8) Å
$V = 5110.1$ (33) Å^3
$Z = 8$
$D_x = 1.312$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ (graphite-monochromated) radiation
$\lambda = 0.71073$ Å
Cell parameters from 33 reflections
$\theta = 9.1$–$14.6°$
$\mu = 0.088$ mm^{-1}
$T = 293$ (2) K
Rectangular
0.40 × 0.40 × 0.35 mm
Colorless

Data collection

Siemens *R*3 diffractometer
ω–2θ scans
Absorption correction: none
15669 measured reflections
4218 independent reflections
2476 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0378$

$\theta_{\rm max} = 29.98°$
$h = -24 \rightarrow 24$
$k = 0 \rightarrow 8$
$l = -27 \rightarrow 27$
3 standard reflections monitored every 100 reflections
intensity decay: 4%

Refinement

Refinement on F^2
$R(F) = 0.0389$
$wR(F^2) = 0.1041$
$S = 0.970$
4218 reflections
343 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.199$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.147$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (5)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.88709 (6)	0.40290 (11)	0.56275 (6)	0.0393 (5)
O2	0.84723 (7)	0.11464 (12)	0.60192 (6)	0.0465 (5)
O3	0.96086 (7)	0.1060 (2)	0.71673 (6)	0.0512 (6)
O4	0.85282 (7)	0.28612 (13)	0.72268 (6)	0.0783 (6)
O10	0.97997 (6)	0.11153 (10)	0.59777 (6)	0.0402 (5)
C1	0.91031 (10)	0.1294 (2)	0.44742 (9)	0.0468 (8)
C2	0.90749 (11)	0.1683 (3)	0.38344 (10)	0.0543 (11)
C3	0.91165 (10)	0.2596 (2)	0.37555 (9)	0.0531 (11)
C4	0.92032 (9)	0.3152 (2)	0.43180 (8)	0.0407 (7)
C4a	0.92255 (8)	0.2764 (2)	0.49633 (8)	0.0295 (8)
C4b	0.93443 (9)	0.3248 (2)	0.56301 (7)	0.0299 (6)
C4c	1.01783 (9)	0.3472 (2)	0.57521 (8)	0.0310 (6)
C5	1.06149 (10)	0.4070 (2)	0.53834 (9)	0.0429 (7)
C6	1.13631 (10)	0.4222 (2)	0.55755 (10)	0.0526 (8)
C7	1.16685 (11)	0.3768 (2)	0.61245 (10)	0.0508 (8)
C8	1.12366 (10)	0.3163 (2)	0.64866 (9)	0.0452 (7)
C8a	1.04795 (9)	0.30121 (15)	0.63016 (8)	0.0329 (6)
C8b	0.98952 (9)	0.2420 (2)	0.66381 (8)	0.0307 (8)
C8c	0.91655 (8)	0.2518 (2)	0.61911 (7)	0.0306 (7)
C8d	0.91268 (9)	0.1612 (2)	0.57989 (8)	0.0371 (7)
C8e	0.91575 (8)	0.1849 (2)	0.50439 (8)	0.0311 (8)
C9	1.00579 (9)	0.1414 (2)	0.66301 (8)	0.0373 (8)
C10	0.84472 (9)	0.2732 (2)	0.66213 (8)	0.0432 (7)
C11	0.76625 (9)	0.28222 (15)	0.63228 (8)	0.0372 (7)
C12	0.70826 (10)	0.3033 (2)	0.67714 (9)	0.0546 (8)
C13	0.63351 (11)	0.3076 (2)	0.65532 (11)	0.0664 (9)
C14	0.61545 (11)	0.2926 (2)	0.58848 (12)	0.0609 (8)
C15	0.67252 (10)	0.2732 (2)	0.54281 (10)	0.0526 (8)
C16	0.74722 (10)	0.2680 (2)	0.56441 (8)	0.0485 (7)
C17	1.08902 (9)	0.1136 (2)	0.67007 (9)	0.0394 (7)
C18	1.13490 (11)	0.1025 (2)	0.61351 (10)	0.0499 (8)
C19	1.21271 (12)	0.0884 (2)	0.62099 (13)	0.0701 (9)
C20	1.24462 (13)	0.0840 (2)	0.68470 (15)	0.0753 (9)
C21	1.19971 (13)	0.0939 (2)	0.74141 (13)	0.0698 (9)
C22	1.12164 (11)	0.1086 (2)	0.73457 (10)	0.0558 (8)
C23	0.89683 (12)	0.4647 (2)	0.61784 (10)	0.0559 (8)
C24	0.83022 (12)	0.0310 (2)	0.57024 (13)	0.0705 (10)
C25	0.95507 (14)	0.0120 (3)	0.71989 (12)	0.0792 (12)

Table 4. *Selected geometric parameters (Å, °) for (5)*

O1—C23	1.428 (2)	C4a—C4b	1.506 (2)
O1—C4b	1.429 (2)	C4b—C4c	1.517 (2)
O2—C8d	1.408 (2)	C4b—C8c	1.577 (3)
O2—C24	1.424 (3)	C4c—C8a	1.381 (2)
O3—C25	1.406 (3)	C8a—C8b	1.503 (3)
O3—C9	1.416 (2)	C8b—C9	1.526 (3)
O4—C10	1.210 (2)	C8b—C8c	1.556 (2)
O10—C9	1.427 (2)	C8c—C10	1.548 (2)
O10—C8d	1.435 (2)	C8c—C8d	1.554 (3)
C1—C8e	1.392 (3)	C8d—C8e	1.522 (2)
C4a—C8e	1.377 (3)	C9—C17	1.521 (3)
C23—O1—C4b	116.92 (13)	C10—C8c—C4b	113.5 (2)
C8d—O2—C24	117.9 (2)	C8d—C8c—C4b	105.24 (14)
C25—O3—C9	116.4 (2)	C8b—C8c—C4b	107.05 (14)
C9—O10—C8d	108.5 (2)	O2—C8d—O10	109.9 (2)
C2—C1—C8e	118.7 (3)	O2—C8d—C8e	116.17 (14)
C8e—C4a—C4	120.9 (2)	O10—C8d—C8e	109.14 (14)
C4—C4a—C4b	126.4 (3)	O2—C8d—C8c	108.18 (14)
O1—C4b—C4a	107.84 (14)	O10—C8d—C8c	106.94 (13)
O1—C4b—C4c	112.3 (2)	C8e—C8d—C8c	106.1 (2)

C4a—C4b—C4c	112.03 (13)	C4a—C8e—C1	120.1 (2)
O1—C4b—C8c	116.67 (13)	C4a—C8e—C8d	110.1 (2)
C4a—C4b—C8c	104.3 (2)	C1—C8e—C8d	129.7 (3)
C4c—C4b—C8c	103.50 (13)	O3—C9—O10	111.9 (2)
C5—C4c—C8a	121.2 (2)	O3—C9—C17	111.3 (2)
C5—C4c—C4b	126.4 (2)	O10—C9—C17	107.5 (2)
C8a—C4c—C4b	112.4 (2)	O3—C9—C8b	104.7 (2)
C4c—C8a—C8b	111.90 (14)	O10—C9—C8b	104.9 (2)
C8—C8a—C8b	129.0 (2)	C17—C9—C8b	116.4 (2)
C8a—C8b—C9	116.4 (2)	O4—C10—C11	118.40 (15)
C8a—C8b—C8c	104.93 (15)	O4—C10—C8c	118.11 (14)
C9—C8b—C8c	103.8 (2)	C11—C10—C8c	123.45 (14)
C10—C8c—C8d	114.40 (15)	C18—C17—C9	121.2 (2)
C10—C8c—C8b	112.37 (13)	C22—C17—C9	119.4 (2)
C8d—C8c—C8b	103.5 (2)		

Crystals of appropriate dimensions of both (3) and (5) were mounted on glass fibers in random orientations. Auto-indexing of ten centered reflections from the rotation photograph indicated a monoclinic lattice for (3) and an orthorhombic lattice for (5). Axial photographs were taken to confirm the Laue symmetry and cell lengths. Intensity data were collected with a variable scan speed. Data reduction, structure solution and structure refinement were carried out using the *SHELXTL-Plus* software package (Sheldrick, 1995). The structures were solved by direct methods. Full-matrix least-squares refinement minimized $w(F_o^2-F_c^2)^2$. The non-H atoms were refined anisotropically to convergence. All H atoms were refined using an appropriate riding model.

For both compounds, data collection: *P*3 (Siemens, 1990); cell refinement: *P*3; data reduction: *XDISK* in *SHELXTL Plus* (Sheldrick, 1995); program(s) used to solve structures: *SHELXTL-Plus*; program(s) used to refine structures: *SHELXTL-Plus*; molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXTL-Plus*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1008). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Photoproduct Derived from 9-Cyano-dibenzobarrelene

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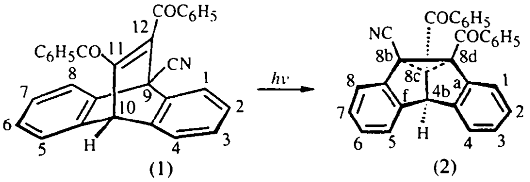
Abstract

The structure of the photoproduct derived from 9-cyano-11,12-dibenzoyl-9,10-dihydro-9,10-ethenoanthracene has been unambiguously established as 8b-cyano-8c,8d-dibenzoyl-4b,8b,8c,8d-tetrahydrodibenzo[*a*,*f*]cyclopropa[*c*,*d*]pentalene, $C_{31}H_{19}NO_2$, through X-ray crystallographic analysis.

Comment

It has been reported previously that dibenzobarrelenes containing the 1,2-dibenzoylalkene moiety undergo photorearrangement on irradiation to give predominantly the corresponding dibenzocyclopropapentalenes or products derived from them (Kumar, Murty, Lahiri, Chackacheri, Scaiano & George 1994; Murty, Pratapan, Kumar, Das & George, 1995; Pratapan, Ashok, Cyr, Das & George, 1987; Pratapan, Ashok, Gopidas, Rath, Das & George, 1990; Asokan, Kumar, Das, Rath & George, 1991; Kumar, Asokan, Das, Wilbur, Rath & George, 1993). If the dibenzobarrelenes have bridgehead substituents, then regioisomeric dibenzocyclopropapentalenes could be formed on irradiation. The regioselectivity in these rearrangements could, however, depend on several factors including the steric and electronic requirements of the bridgehead substituent. We have reported previously (Murty, Pratapan, Kumar, Das & George, 1995) that the photolysis of 9-cyano-11,12-dibenzoyl-9,10-dihydro-9,10-ethanoanthracene, (1), gives exclusively the title 8b-cyanosubstituted dibenzocyclopropapentalene, (2) and not the 4b-substituted regioisomer.



(1) (2)

In view of the recent report that dibenzocyclopropapentalenes containing three substituents attached to the cyclopropane ring show a propensity for ring