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10-(3-Methoxybenzylidene)anthrone

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.009 Å
R factor = 0.053
wR factor = 0.184
Data-to-parameter ratio = 7.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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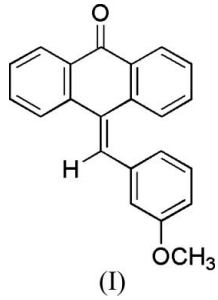
The title compound, $C_{22}H_{16}O_2$, was prepared from anthrone and 3-methoxybenzaldehyde in the presence of pyridine. X-ray analysis shows that the anthraquinone ring system is not planar. The central six-membered ring adopts an asymmetric boat conformation, in which the carbonyl C and the *para*-C atoms deviate from the plane of the other four atoms by 0.162 (8) and 0.267 (8) Å, respectively. Intermolecular C—H···O hydrogen bonds are present in the crystal structure.

Comment

10-Substituted benzylideneanthrones have been known for a long time for their widespread use as functional and disperse dyes (Day, 1963). Recently, however, some 10-substituted benzylideneanthrones have been found to possess high antitumor activity (Paull *et al.*, 1992; Prinz *et al.*, 2003). Owing to our interest in this area, we have prepared a series of 10-substituted benzylideneanthrones in our laboratory and evaluated their anticancer activity. Our study on structure–activity relationships showed that substitution of the benzylidene aromatic ring affects the antitumor activity (Hu & Zhou, 2004). In a continuation of this research, we have prepared the title compound, (I), and investigated its crystal structure.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Selected bond lengths and angles are listed in Table 1. The three rings of the anthraquinone system are not coplanar, the two outer benzene rings forming a dihedral angle of 22.0 (2)°. The central ring adopts an asymmetric boat conformation; atoms C5 and C10 deviate from the plane of the other four atoms by 0.267 (8) and 0.162 (8) Å, respectively. Fig. 2 shows that each molecule is involved in two intermolecular hydrogen bonds (Table 2), which contribute to the formation of the crystal structure.

Experimental

To a mixture of anthrone (4.0 g, 21 mmol) and 3-methoxybenzaldehyde (5.0 g, 37 mmol) were added pyridine (30 ml) and

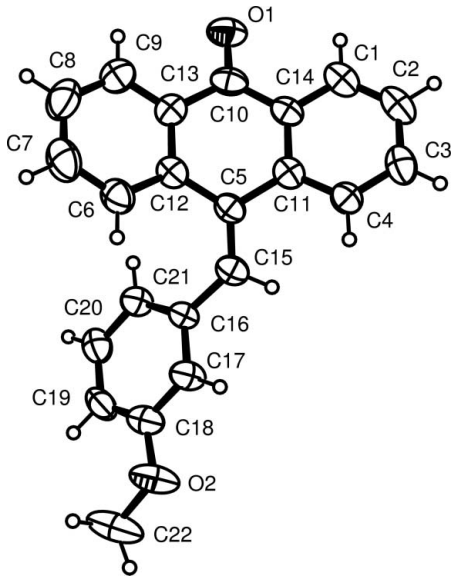


Figure 1
The structure of (I), with 50% probability displacement ellipsoids.

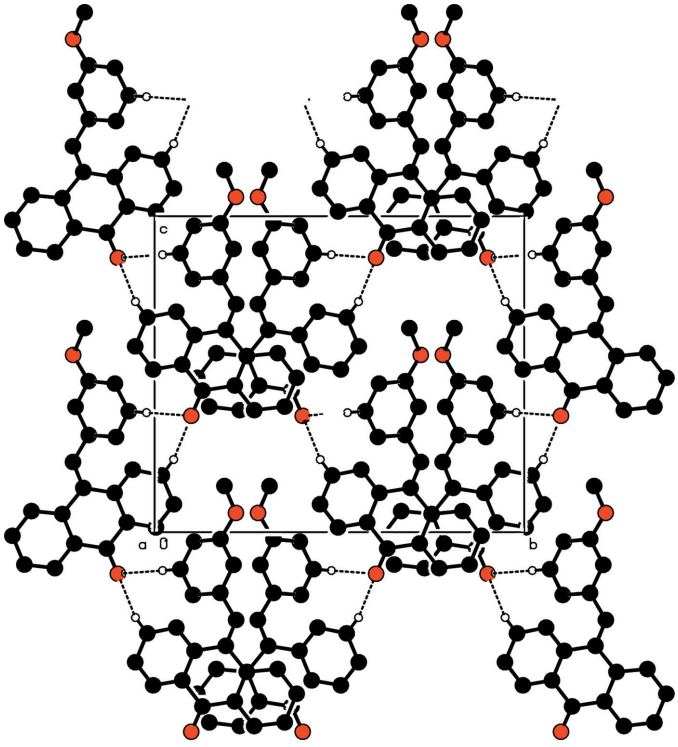


Figure 2
Packing diagram of (I), viewed along the *a* axis, showing hydrogen bonds as dashed lines. For clarity, H atoms have been omitted, except for those involved in hydrogen bonding.

piperidine (0.5 g, 6 mmol). The reaction mixture was refluxed under nitrogen for 6 h until the reaction was complete, as evidenced by thin-layer chromatography. The mixture was cooled to room temperature and poured into methanol (100 ml); it was then placed in a refrigerator overnight. The precipitate was filtered off and washed with methanol to afford yellow crystals (4.7 g, yield 75.3%, m.p. 386–388 K). A few crystals were dissolved in absolute ethanol, which was allowed to evaporate slowly to give yellow crystals of (I) suitable for X-ray diffraction studies.

Crystal data

$C_{22}H_{16}O_2$
M_r = 312.35
Orthorhombic, $Pna2_1$
a = 7.165 (2) Å
b = 16.345 (2) Å
c = 13.963 (3) Å
V = 1635.2 (6) Å^3
Z = 4
D_x = 1.269 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.5–10.8°
μ = 0.08 mm^{-1}
T = 295 (2) K
Block, yellow
0.40 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.963, T_{max} = 0.967
1647 measured reflections
1532 independent reflections
818 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.2°
$h = 0 \rightarrow 8$
$k = -19 \rightarrow 1$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.184
S = 1.05
1532 reflections
218 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1119P)^2 + 0.1776P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C10	1.238 (7)	C10—C14	1.447 (9)
C5—C15	1.354 (8)	C10—C13	1.492 (8)
C5—C12	1.462 (8)	C11—C14	1.409 (8)
C5—C11	1.483 (8)	C12—C13	1.412 (9)
C15—C5—C12	124.6 (5)	C17—C16—C15	119.9 (5)
C15—C5—C11	118.3 (5)	C21—C16—C15	121.9 (5)
C5—C15—C16	131.5 (6)		
C11—C5—C12—C13	23.0 (8)	C5—C11—C14—C10	5.7 (7)
C5—C12—C13—C10	−3.8 (8)	C12—C5—C15—C16	9.2 (10)
C14—C10—C13—C12	−14.8 (8)	C5—C15—C16—C21	39.7 (9)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1^{i}	0.93	2.40	3.298 (10)	163
C20—H20···O1^{ii}	0.93	2.51	3.441 (8)	174

Symmetry codes: (i) $-x, -y+1, z+\frac{1}{2}$; (ii) $-x+1, -y+1, z+\frac{1}{2}$.

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.96 Å for methyl H atoms and 0.93 Å for other H atoms; U_{iso}(H) values were set at 1.2 (1.5 for methyl) times U_{eq}(carrier atom). In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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