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10-(3,4-Dimethoxybenzylidene)anthrone

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.110
Data-to-parameter ratio = 13.2

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

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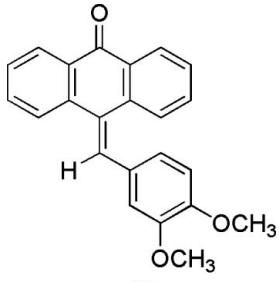
The title compound, $C_{23}H_{18}O_3$, was prepared from anthrone and 3,4-dimethoxybenzaldehyde in the presence of pyridine. X-ray analysis shows that the three rings of the anthraquinone moiety are not coplanar; the central six-membered ring assumes an asymmetric boat conformation in which the carbonyl C and the opposite C atom deviate from the plane by 0.190 (3) and 0.262 (3) Å, respectively.

Comment

10-Substituted benzylideneanthrones have been known for a long time for their widespread use as functional dyes 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 and evaluated their anticancer activity. Our study on the structure–activity relationship (SAR) showed that a substituent on the benzylidene part of the molecule can affect the antitumor activity (Hu & Zhou, 2004). In a continuation of our research on SAR, we prepared the title compound, (I), and investigated its structure.



(I)

The molecular structure of (I) is illustrated in Fig. 1, and selected bond lengths and angles are listed in Table 1. The three rings of the anthraquinone moiety are not coplanar, the dihedral angle between the two outer benzene rings being 24.68 (9)°. In the central six-membered ring, atoms C11/C12/C13/C14 are coplanar within 0.0083 (9) Å, atoms C5 and C10 deviating from the plane by 0.262 (3) and 0.190 (3) Å, respectively; the ring assumes an asymmetric boat conformation.

Experimental

To a mixture of anthrone (2.0 g, 10 mmol) and 3,4-dimethoxybenzaldehyde (2.0 g, 12 mmol) were added pyridine (30 ml) and piperidine (0.5 g, 6 mmol). The reaction mixture was refluxed for 6 h

and thin-layer chromatography showed that the reaction was complete. The mixture was cooled to room temperature, poured into methanol (75 ml) and placed in a refrigerator overnight. The precipitate which formed was collected and recrystallized twice from glacial acetic acid to afford orange crystals of (I) (2.4 g, yield 70.2%, m.p. 453–456 K). Crystals suitable for X-ray analysis were grown from an ethanol solution by slow evaparation.

Crystal data

$C_{23}H_{18}O_3$
M_r = 342.37
Monoclinic, $P2_1/n$
a = 11.709 (3) Å
b = 10.306 (2) Å
c = 14.847 (6) Å
β = 103.36 (3)°
V = 1743.1 (9) Å^3
Z = 4
D_x = 1.305 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.7–14.0°
μ = 0.09 mm^{-1}
T = 295 (2) K
Prism, orange
0.40 × 0.40 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3529 measured reflections
3136 independent reflections
1832 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.2°
$h = -14 \rightarrow 13$
$k = 0 \rightarrow 12$
$l = -1 \rightarrow 17$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.110
S = 0.99
3136 reflections
238 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 0.3183P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0133 (12)

Table 1
Selected geometric parameters (Å, °).

O1—C10	1.230 (2)	C10—C14	1.467 (3)
C5—C15	1.348 (2)	C10—C13	1.471 (3)
C5—C12	1.480 (2)	C11—C14	1.403 (2)
C5—C11	1.484 (3)	C12—C13	1.405 (2)
C15—C5—C12	124.86 (17)	C21—C16—C15	123.32 (17)
C15—C5—C11	119.09 (16)	C17—C16—C15	118.17 (18)
C5—C15—C16	131.69 (18)		
C11—C5—C12—C13	21.8 (2)	C5—C11—C14—C10	4.7 (3)
C5—C12—C13—C10	−1.5 (3)	C12—C5—C15—C16	6.5 (3)
C14—C10—C13—C12	−17.5 (3)	C5—C15—C16—C21	48.2 (3)

H atoms were placed at calculated positions and refined using a riding model. H atoms were given isotropic displacement parameters equal to $1.2U_{eq}$ of their parent atoms (1.5 for methyl H atoms) and C—H distances were constrained to 0.93 Å (0.96 Å for methyl H atoms).

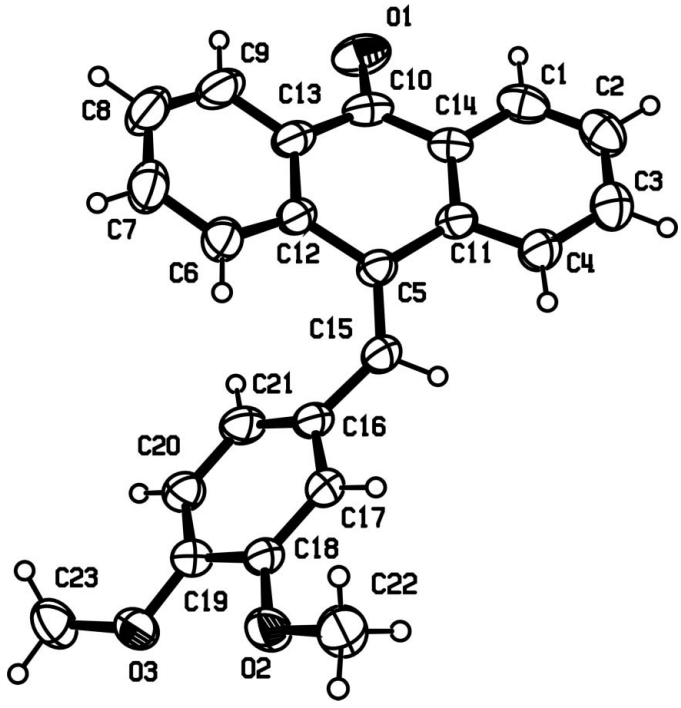


Figure 1
The structure of (I), shown with 50% probability displacement ellipsoids.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD* (McArdle, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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