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10-(5-Chloro-2-hydroxybenzylidene)anthrone acetonitrile solvate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.050
wR factor = 0.159
Data-to-parameter ratio = 13.6

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

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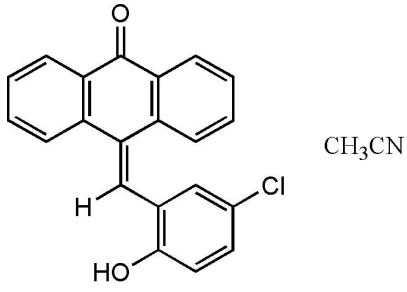
The title compound, $C_{21}H_{13}ClO_2\cdot CH_3CN$, was prepared from anthrone and 5-chloro-2-hydroxybenzaldehyde and recrystallized from acetonitrile. The central six-membered ring has an asymmetric boat conformation, in which the carbonyl C atom and the opposite C atom deviate from the plane of the other four atoms by 0.170 (5) and 0.302 (5) Å, respectively.

Comment

It was reported recently that some 10-substituted benzylideneanthrones possess high antitumour activity (Paull *et al.*, 1992). In our laboratory, some 10-substituted benzylideneanthrones have been prepared and evaluated for antitumour activity. Our study of structure–activity relationships (SAR) showed that substituents on the phenyl ring of the molecule affect its antitumour activity (Hu & Zhou, 2004). As a continuation of our research work on SAR, we have prepared crystals of the title compound, (I), and investigated its structure.



(I)

The molecular structure of (I) is illustrated in Fig. 1, and consists of discrete molecules of 10-(5-chloro-2-hydroxybenzylidene)anthrone and acetonitrile solvent in a 1:1 ratio. Selected bond lengths and angles are listed in Table 1. Atoms C11, C12, C13 and C14 are coplanar to within 0.0072 (19) Å, while atoms C5 and C10 deviate from the plane by 0.302 (5) and 0.170 (5) Å, respectively.

Experimental

To a mixture of anthrone (2.0 g, 10 mmol) and 5-chloro-2-hydroxybenzaldehyde (1.9 g, 12 mmol) were added pyridine (30 ml) and piperidine (0.5 g, 6 mmol). The reaction mixture was refluxed for 6 h. Testing by thin-layer chromatography showed complete reaction of the anthrone. The mixture was cooled to room temperature and poured into methanol (75 ml), and then put in a refrigerator overnight. The resulting precipitate was collected and recrystallized from benzene to afford yellow crystals of (I) (1.5 g, yield 45.1%; m.p. 468–

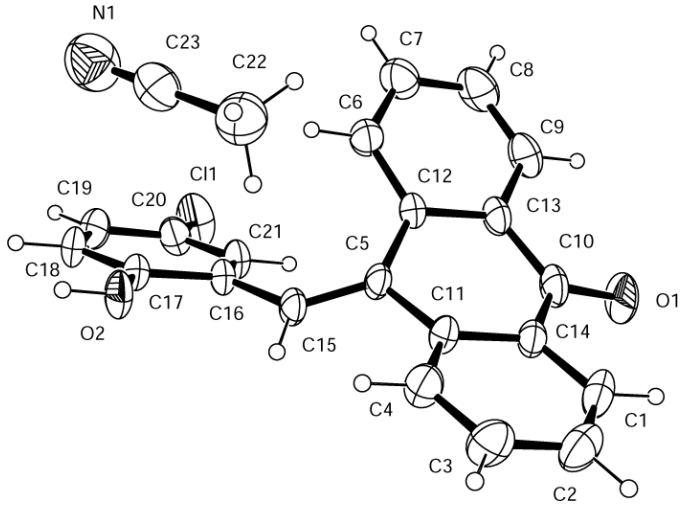


Figure 1
A view of the structure of (I), with the atom-numbering scheme. Dipslacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

469 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3358, 1646, 1596, 1490, 1474, 1411 1324, 1276, 933, 814, 775, 685; ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.45 (*s*, 1H, C═CH), 5.08 (*s*, 1H, OH), 6.82–8.30 (*m*, 11H). As this crystalline product was not found to be suitable for X-ray diffraction studies, a few crystals were dissolved in acetonitrile, which was allowed to evaporate slowly to give brown crystals of (I) suitable for X-ray structure studies.

Crystal data

$C_{21}H_{13}ClO_2{\cdot}C_2H_3N$
M_r = 373.82
Monoclinic, $P2_1/c$
a = 7.602 (2) Å
b = 23.090 (6) Å
c = 10.711 (3) Å
β = 92.05 (2)°
V = 1878.9 (9) Å^3
Z = 4
D_x = 1.321 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9.6–11.1°
μ = 0.22 mm^{-1}
T = 295 (2) K
Plate, brown
0.50 × 0.35 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.898, T_{max} = 0.986
3746 measured reflections
3368 independent reflections
2241 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.2°
$h = -9 \rightarrow 9$
$k = -1 \rightarrow 27$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.159
S = 0.98
3368 reflections
247 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0095 (16)

Table 1
Selected geometric parameters (Å, °).

Cl1—C20	1.732 (4)	O2—C17	1.358 (4)
O1—C10	1.237 (4)	C5—C15	1.348 (4)
C15—C5—C12	124.7 (3)	C21—C16—C15	122.8 (3)
C15—C5—C11	118.9 (3)	C17—C16—C15	119.4 (4)
C12—C5—C15—C16	5.5 (6)	C5—C15—C16—C21	39.1 (6)
C11—C5—C15—C16	−169.7 (4)	C5—C15—C16—C17	−144.7 (4)

H atoms were included at calculated positions and refined using a riding model, with an O—H distance of 0.83 Å and C—H distances of 0.96 Å for methyl H and 0.93 Å for all others, and with U_{iso}(H) = 1.2U_{eq}(C,O), or 1.5U_{eq}(C) for methyl H atoms.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Version 1.05; Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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