Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

10-(4-Hydroxy-3-nitrobenzylidene)anthrone

Wei Zhou, Wei-Xiao Hu* and Chun-Nian Xia

College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou, 310032, People's Republic of China

Correspondence e-mail: huyang@mail.hz.zj.cn

Received 3 November 2006
Accepted 16 November 2006

The title compound, $C_{21}H_{13}NO_4$, was prepared by the reaction of anthrone and 4-hydroxy-3-nitrobenzaldehyde catalysed by anhydrous hydrogen chloride. In the molecule, the anthraquinone fragment is non-planar; the central six-membered ring adopts an asymmetric boat conformation, while the two outer rings make a dihedral angle of 36.0 (1)°. Weak intermolecular C—H···O hydrogen bonds link the molecules into centrosymmetric dimers.

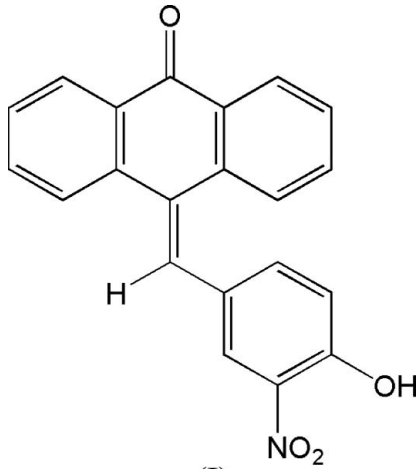
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.137
Data-to-parameter ratio = 11.6

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

Comment

10-Substituted benzylideneanthrones are widely used as functional and disperse dyes (Day, 1963). Recently, high antitumor activity has been reported (Paull *et al.*, 1992; Helge *et al.*, 2003) for some of these compounds. As a result of our interest in this area, we have prepared a series of 10-substituted benzylideneanthrones and evaluated their anticancer activity. Our study of structure–activity relationships (SAR) showed that the substitutent on the phenyl ring of the molecule affects its antitumor activity (Hu & Zhou, 2004). In a continuation of our SAR investigations, we present here the crystal structure of the title compound, (I).



(I)

In (I) (Fig. 1), the three rings of the anthraquinone fragment are not coplanar. The central six-membered ring adopts an asymmetric boat conformation, with atoms C5 and C10 deviating from the C11–C14 mean plane [r.m.s. deviation = 0.017 (1) Å] by 0.352 (4) and 0.253 (4) Å, respectively. The two outer rings make a dihedral angle of 36.0 (1)°. The mean planes C1–C5/C11/C14 and C15–C21 make a dihedral angle of 42.83 (9)°. The hydroxy group is involved in the formation of an intramolecular O—H···O hydrogen bond (Table 1).

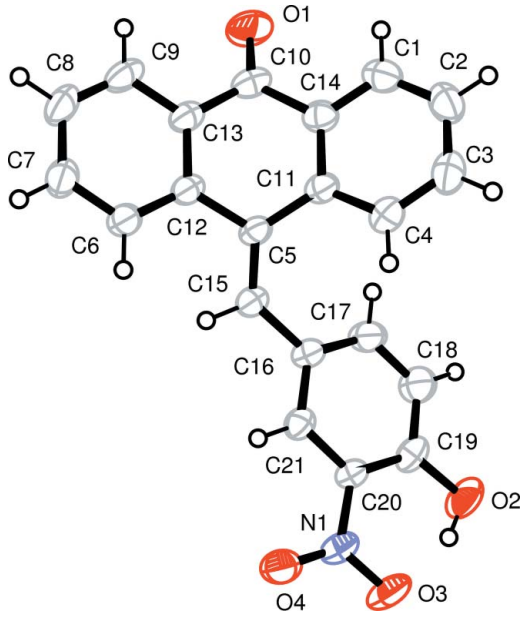


Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids.

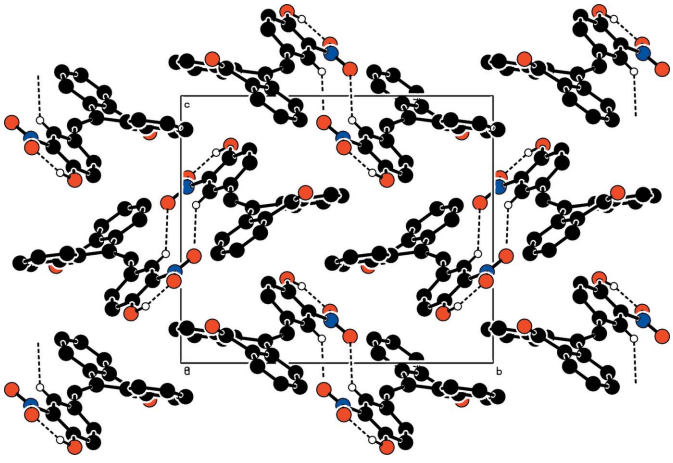


Figure 2
A packing diagram, viewed along the *a* axis, showing the hydrogen bonds as dashed lines. For clarity, H atoms have been omitted unless involved in hydrogen bonding.

In the crystal structure (Fig. 2), weak intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into centrosymmetric dimers.

Experimental

Through a mixture of anthrone (1.6 g, 8.2 mmol) and 4-hydroxy-3-nitrobenzaldehyde (1.4 g, 8.4 mmol) in 25 ml absolute alcohol was slowly bubbled anhydrous hydrogen chloride. The reaction mixture was heated at 343 K until thin-layer chromatography showed that the reaction was complete. The precipitate was then filtered off, washed with absolute ethanol, and recrystallized from absolute ethanol to give orange crystals of the compound (yield 2.7 g, 93.4%; m.p. 467–469 K). Since these crystals were of poor quality, a number of them were dissolved in absolute ethanol, which was evaporated slowly to give orange crystals of (I) suitable for X-ray diffraction.

Crystal data

$C_{21}H_{13}NO_4$
M_r = 343.32
Monoclinic, $P2_1/c$
a = 8.545 (4) Å
b = 14.588 (3) Å
c = 13.916 (4) Å
β = 116.22 (2)°
V = 1556.3 (9) Å^3
Z = 4
D_x = 1.465 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
Prism, orange
0.50 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.951, T_{max} = 0.980
3238 measured reflections
2790 independent reflections
1590 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 25.2°
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.137
S = 1.04
2790 reflections
240 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.1995P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.011 (2)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*X*···O3	0.86 (4)	1.78 (3)	2.569 (3)	153 (4)
C21—H21···O4^{i}	0.93	2.56	3.314 (3)	138

Symmetry code: (i) $-x+1, -y-1, -z+2$.

C-bound H atoms were placed at calculated positions and refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). The hydroxy H atom was located in a difference map and refined isotropically with the restraint O–H = 0.85 (2) Å.

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: *ORTEP-3 for Windows* (Version 1.05; Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

We acknowledge the support of the Science and Technology Development Fund of Zhejiang University of Technology.

References

Day, J. H. (1963). *Chem. Rev.* **63**, 65–80.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Helge, P., Yukihito, I., Takeo, H. & Thomas, S. (2003). *J. Med. Chem.* **46**, 3382–3394.

Hu, W. X. & Zhou, W. (2004). *Bioorg. Med. Chem. Lett.* **14**, 621–622.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Paull, K. D., Lin, C. M., Malspeis, L. & Hamel, E. (1992). *Cancer Res.* **52**, 3892–3900.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.