Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

10-(3-Acetamidobenzylidene)anthrone

Wei Zhou,[a] Wei-Xiao Hu,[a]* Chun-Nian Xia[a] and Min-Qin Chen[b]

[a]College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China, and [b]Fudan University, Shanghai 200433, People's Republic of China

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

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.007 Å
R factor = 0.051
wR factor = 0.150
Data-to-parameter ratio = 9.0

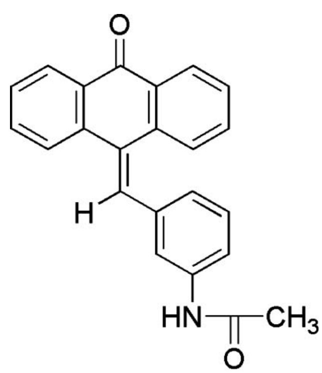
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{23}H_{17}NO_2$, was prepared by reacting 10-(3-aminobenzylidene)anthrone and acetic anhydride in the presence of pyridine. X-ray crystal structure analysis shows that the three rings of the anthraquinone system are not coplanar because of steric interactions with the benzylidene group.

Received 19 August 2005
Accepted 5 September 2005
Online 14 September 2005

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 the structure–activity relationship (SAR) showed that substitution in the phenyl ring of the molecule affects its antitumour activity (Hu & Zhou, 2004). As a continuation of our research work on SARs, we prepared crystals of the title molecule, (I), and investigated its structure.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Selected bond lengths and angles are listed in Table 1. As illustrated in Fig. 1, the three rings of the anthraquinone system are not coplanar, because of steric interactions with the benzylidene group; the two outer benzene rings form a dihedral angle of 23.0 (1)°. In the central six-membered ring, atoms C11, C12, C13 and C14 are coplanar to within 0.009 (2) Å, with atoms C5 and C10 deviating from the plane by 0.125 (5) and 0.246 (5) Å, respectively, and the ring adopts an asymmetric boat conformation.

Experimental

10-(3-Aminobenzylidene)anthrone was prepared according to the literature method of Ingram (1950). A mixture of 10-(3-aminobenzylidene)anthrone (0.6 g), acetic anhydride (20 ml) and pyridine

(0.5 ml) was added to a 100 ml flask and then stirred. The reaction was continued for 1.5 h at room temperature, followed by filtration and washing with ethanol. The crude product was collected and recrystallized from ethanol to afford yellow crystals of (I) (0.4 g, yield 59.0%, m.p. 462–465 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3319, 1687, 1645, 1602, 1544, 1483, 1417, 1372, 1319, 1277, 932, 776, 683; ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.52 (*s*, 1H, C=CH), 7.00–8.28 (*m*, 12H), 2.18 (*s*, 3H, $-CH_3$). MS (%): 340 (*M*+1, 10), 339 (*M*, 22), 297 (11), 296 (32), 281 (17), 280 (47), 239 (10), 43 (100).

Crystal data

$C_{23}H_{17}NO_2$	D_x = 1.264 Mg m^{-3}
M_r = 339.38	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 25 reflections
a = 12.3847 (3) Å	θ = 3.3–27.5°
b = 7.4853 (4) Å	μ = 0.08 mm^{-1}
c = 12.3351 (3) Å	T = 296 (2) K
β = 128.764 (4)°	Block, yellow
V = 891.63 (8) Å^3	0.34 × 0.23 × 0.14 mm
Z = 2	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.029
ω–2θ scans	θ_{max} = 27.5°
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	h = −16 → 0
T_{min} = 0.963, T_{max} = 0.989	k = −9 → 9
3717 measured reflections	l = −11 → 16
2171 independent reflections	3 standard reflections frequency: 60 min intensity decay: none
1626 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0888P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.051	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.151	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
S = 1.10	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
2171 reflections	Extinction correction: *SHELXL97* (Sheldrick, 1997)
241 parameters	Extinction coefficient: 0.084 (12)
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °) for (I).

O1—C5	1.231 (4)	N1—C18	1.408 (5)
O2—C22	1.219 (4)	C10—C15	1.350 (4)
N1—C22	1.354 (4)		
C15—C10—C14	118.5 (3)	C17—C16—C21	119.0 (3)
C15—C10—C13	125.0 (3)	C17—C16—C15	122.5 (3)
C14—C10—C15—C16	−176.1 (4)	C10—C15—C16—C21	−127.7 (4)
C13—C10—C15—C16	2.9 (6)	C22—N1—C18—C19	−22.1 (6)
C10—C15—C16—C17	55.5 (6)	C22—N1—C18—C17	159.2 (4)

H atoms were positioned geometrically, with C—H distances set to 0.96 Å for methyl H atoms and 0.93 Å for the remainder, and refined using the riding-model approximation, with U_{iso}(H) = 1.2 (or 1.5 for methyl H atoms) times U_{eq}(parent atom). In the absence of significant anomalousn scattering, Friedel pairs were merged before the final refinement .

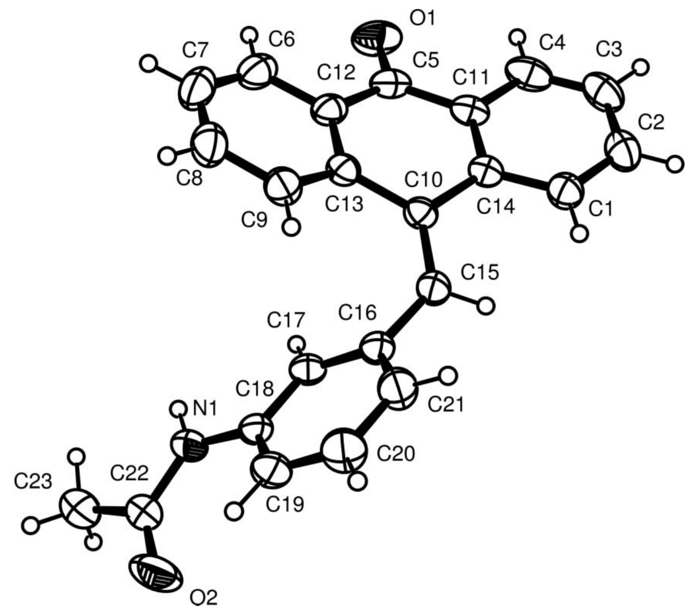


Figure 1
The structure of (I), with 30% probability displacement ellipsoids.

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

We are grateful to the National Natural and Scientific Foundation (grant No. 20272053) for financial support. We also acknowledge the support of the Science and Technology Development Fund (2005) of Zhejiang University of Technology.

References

Enraf–Nonius (1994). *CAD-4 EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Hu, W. X. & Zhou, W. (2004). *Bioorg. Med. Chem. Lett.* **14**, 621–622.
Ingram, V. M. (1950). *J. Chem. Soc.* pp. 2318–2324.
McArdle, P. (2000). *J. Appl. Cryst.* **33**, 993.
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.