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10-(2-Methylbenzylidene)anthrone

Wei Zhou, Wei-Xiao Hu* and Guo-Wu Rao

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

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

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.171
Data-to-parameter ratio = 16.4

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

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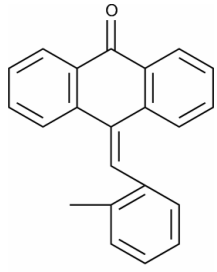
The title compound, $C_{22}H_{16}O$, was prepared from anthrone and 2-methylbenzaldehyde. The central six-membered ring has an asymmetric boat conformation, in which the carbonyl carbon and the opposite carbon deviate from the plane of the ring by 0.124 (3) and 0.283 (2) Å, respectively.

Comment

It was reported recently that the derivatives of 10-substituted benzylideneanthrone have a high potential for antitumor activity (Paull *et al.*, 1992). In a continuation of our work on the structure–activity relationship of the derivatives of 10-substituted benzylidene anthrone (Hu & Zhou, 2004), we have obtained a yellow crystalline compound that was the product of the reaction of anthrone and 2-methylbenzaldehyde. The structural identity of the product, (I), was determined by single-crystal X-ray diffraction.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Selected bond lengths and angles are listed in Table 1. In (I), atoms C1, C6, C8 and C13 are coplanar within 0.0058 (7) Å, with atoms C7 and C14 deviating from the plane by 0.124 (5) and 0.283 (2) Å, respectively. Therefore, the central six-membered ring of (I), has an asymmetric boat conformation.

Experimental

To a mixture of anthrone (3.9 g, 20 mmol) and 2-methylbenzaldehyde (3.0 g, 25 mmol) were added pyridine (30 ml) and piperidine (0.5 g, 6 mmol). The air in the system was removed using an aspirator, and replaced by nitrogen gas; this operation was repeated three times. Nitrogen gas was bubbled through the mixture continuously until the reaction was complete. The reaction mixture was refluxed for 6 h. The completion of the reaction of anthrone was determined by thin-layer chromatography. The mixture was cooled to room temperature and poured into methanol (75 ml) and then placed in a refrigerator overnight. The precipitate was collected and recrystallized twice from acetic acid to afford yellow chunks (0.8 g, yield 14.0%; m.p. 363–366 K).

Crystal data

$C_{22}H_{16}O$
M_r = 296.35
Triclinic, $P\overline{1}$
a = 7.0137 (2) Å
b = 10.3135 (4) Å
c = 11.5107 (5) Å
α = 107.020 (5)°
β = 91.224 (4)°
γ = 100.854 (8)°
V = 779.33 (6) Å^3
Z = 2
D_x = 1.263 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4435 reflections
θ = 2.4–27.4°
μ = 0.08 mm^{-1}
T = 293 (2) K
Chunk, yellow
0.41 × 0.31 × 0.28 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.972, T_{max} = 0.979
5264 measured reflections
3438 independent reflections
2503 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.171
S = 1.09
3438 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1018P)^2 + 0.0246P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.061 (10)

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.2143 (18)	C14—C15	1.339 (2)
O1—C7—C8	121.85 (15)	C14—C15—C16	129.89 (15)
O1—C7—C6	120.93 (16)	C17—C16—C15	119.64 (15)
C15—C14—C1	123.81 (15)	C21—C16—C15	120.74 (15)
C15—C14—C13	119.85 (15)		
C1—C6—C7—C8	−10.1 (2)	C6—C1—C14—C13	25.4 (2)
C6—C7—C8—C13	11.4 (2)	C8—C13—C14—C1	−24.1 (2)

H atoms were positioned geometrically (0.96 Å for methyl H atoms and 0.93 Å for the remainder) and refined using the riding-model approximation, with U_{iso} = 1.2 (or 1.5 for methyl H atoms) times U_{eq}(parent atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

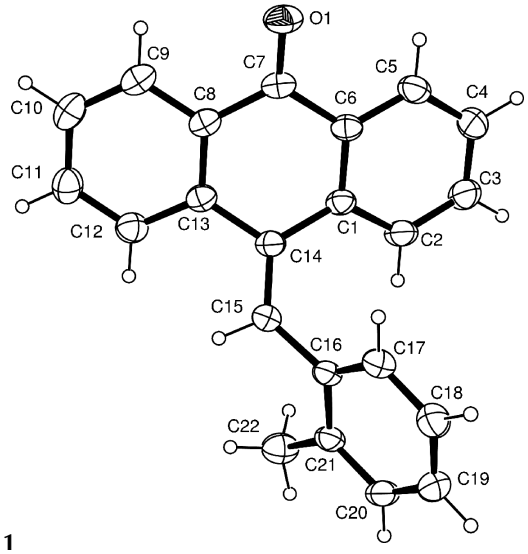


Figure 1
The structure of (I), shown with 30% probability displacement ellipsoids.

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