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5a,6,11*a*,12-Tetrahydro-5a,11*a*-dimethyl-1,4-benzoxazino[3,2-*b*][1,4]benzoxazine

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The title compound, $C_{16}H_{16}N_2O_2$, was prepared by reaction of 2-aminophenol and diacetyl. A colorless crystal was obtained by recrystallization from an MeOH–H_2O solution. X-ray analysis reveals that the molecule has no crystallographically imposed symmetry. The heterocyclic rings adopt opposite twist conformations, leading to an overall V-shaped twist conformation. Weak intermolecular N—H···O hydrogen-bonding interactions link the molecules into a one-dimensional chain along the *c* axis.

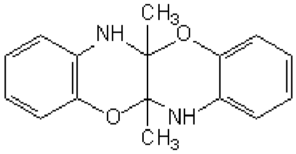
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.077
wR factor = 0.220
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.

Comment

It has been shown by X-ray analysis (Tauer *et al.*, 1986) that the condensation product of 2-aminophenol and glyoxal is 5a,6,11a,12-tetrahydro-1,4-benzoxazino[3,2-*b*][1,4]benzoxazine and not 2,2′-bibenzoxazoline, as generally assumed. This compound has interesting photochemical properties which depend on the wavelength of the exciting light (Tauer & Grellmann, 1990). Some derivatives of this compound, such as 5a,6,11a,12-tetrahydro-5a-methyl-1,4-benzoxazino[3,2-*b*]-[1,4]benzoxazine, have the same properties (Tauer *et al.*, 1986). The crystal structures of 5a,6,11a,12-tetrahydro-5a-methyl-11a-phenyl-1,4-benzoxazino[3,2-*b*][1,4]benzoxazine and other derivatives have been reported (Barluenga *et al.*, 1986). In order to study further the relationship between the crystal structures and the photochemical properties of this type of compound, we report the synthesis and crystal structure of 5a,6,11a,12-tetrahydro-5a,11a-dimethyl-1,4-benzoxazino[3,2-*b*][1,4]benzoxazine, (I).



(I)

A perspective view of the crystal structure and packing diagram of (I) are shown in Figs. 1 and 2, respectively. As can be seen in Fig. 1, the molecule has no crystallographic symmetry, the heterocyclic rings adopting opposite twist conformations, which leads to an overall V-shaped twist conformation. The bond lengths N1—C3, N1—C6, N2—C2 and N2—C12 (Table 1) correspond to single C—N bonds. The bond lengths O1—C2, O1—C5, O2—C3 and O2—C11 are in the range 1.388 (3)–1.480 (3) Å, corresponding to the single C—O bond length. The torsion angles N2—C2—C3—N1 and O1—C2—C3—O2 are −172.47 (18) and 59.7 (3)°, respec-

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tively. In respect of these geometrical parameters, the structure of the title compound is similar to those published by Barluenga *et al.* (1986).

The crystal packing (Fig. 2) is stabilized by weak intermolecular N—H···O hydrogen-bonding interactions (Table 2), which link the molecules into a one-dimensional chain along the *c* axis.

Experimental

2-Aminophenol (46 mmol) and diacetyl (23 mmol) were stirred in methanol (70 ml) for 2 h. The deep-brown solution was allowed to stand in the cold overnight and the product then removed by filtration. It was recrystallized from ethanol with the aid of decolorizing carbon (yield 2.8 g, 49%; m.p. 514–515 K). Spectroscopic analysis, IR (KBr, ν cm^{-1}): 3344, 3032, 2938, 1700, 1570, 1504; ^{1}H NMR (CDCl$_3$, p.p.m.): 1.5 (*s*, 2 CH$_3$), 6.6–6.8 (*m*, 8H aromatic), 7.2 (*s*, 2NH); analysis calculated for $C_{16}H_{16}N_2O_2$: C 71.64, H 5.97, N 10.45%; found: C 70.56, H 4.90, N 10.38%.

Crystal data

$C_{16}H_{16}N_2O_2$
$M_r = 268.31$
Monoclinic, $P2_1/c$
$a = 11.025$ (7) Å
$b = 11.238$ (7) Å
$c = 11.666$ (7) Å
$\beta = 105.484$ (10)°
$V = 1392.9$ (15) Å^3
$Z = 4$

$D_x = 1.279$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 739 reflections
$\theta = 3.6$–25.1°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.20 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
6838 measured reflections
2408 independent reflections

1621 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\rm max} = 25.0°$
$h = -13 \rightarrow 11$
$k = -13 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.077$
$wR(F^2) = 0.220$
$S = 1.01$
2408 reflections
183 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1574P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.72$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C6	1.398 (4)	O1—C5	1.390 (3)
N1—C3	1.412 (3)	O1—C2	1.480 (3)
N2—C12	1.387 (4)	O2—C11	1.388 (3)
N2—C2	1.402 (3)	O2—C3	1.477 (3)
C6—N1—C3	118.5 (2)	N2—C2—C3	110.4 (2)
C12—N2—C2	118.6 (2)	O1—C2—C3	107.34 (18)
C5—O1—C2	117.3 (2)	N1—C3—O2	106.42 (19)
C11—O2—C3	116.8 (2)	N1—C3—C4	110.8 (2)
N2—C2—O1	107.15 (19)	O2—C3—C4	107.9 (2)
N2—C2—C1	111.1 (2)	N1—C3—C2	110.5 (2)
O1—C2—C1	107.8 (2)	O2—C3—C2	107.48 (19)
N2—C2—C3—N1	−172.47 (18)	O1—C2—C3—O2	59.7 (3)

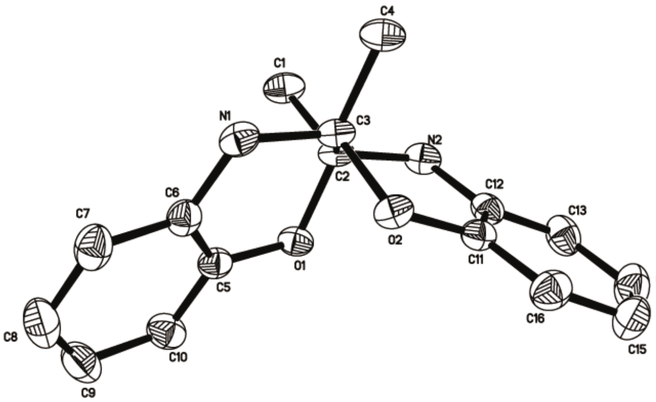

Figure 1
A view of the title compound, showing 30% probability displacement ellipsoids. H atoms have been omitted.

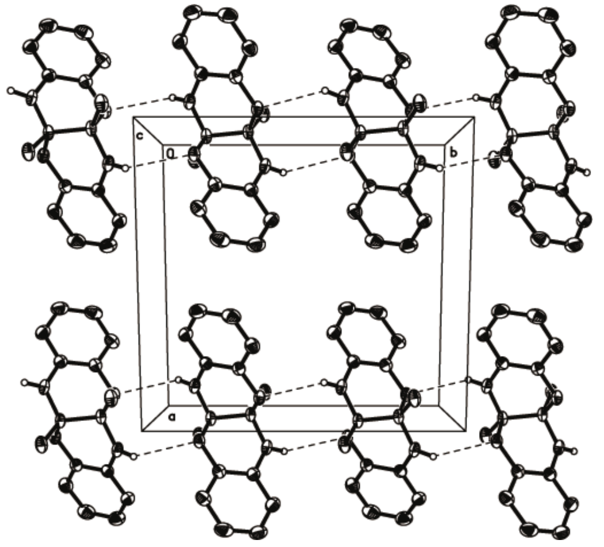

Figure 2
Packing diagram of the title compound viewed down the *b* axis. Only H atoms bonded to N atoms are shown. Hydrogen bonds are represented by dashed lines.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2$^{\rm i}$	0.86	2.55	3.122 (4)	125
N2—H2···O1$^{\rm ii}$	0.86	2.54	3.117 (3)	126

Symmetry codes: (i) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $-x, \frac{3}{2}+y, \frac{1}{2}-z$.

H atoms were included in calculated positions and refined with fixed displacement parameters, riding on their parent atoms, with C—H distances in the range 0.93–0.98 Å, N—H distances in the range 0.86−0.90 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(C,N). The relatively high maximum residual density is situated 1.2 Å from atom N2.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-NT* (Bruker, 1998); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-NT* (Bruker, 1998); software used to prepare material for publication: *SHELXTL-NT*.

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