

Jia-Ning Xu, Jin-Peng Zhang,
Qian Wang, Xiao-Tong Zhu and
Shu-Jiang Tu*Department of Chemistry, Xuzhou Normal
University, Xuzhou 221116, People's Republic
of China

Correspondence e-mail: laotu2001@263.net

Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.037
 wR factor = 0.091
Data-to-parameter ratio = 12.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.9-(4-Bromophenyl)-3,3,6,6-tetramethyl-3,4,5,6,9,10-
hexahydroacridine-1,8(2H,7H)-dione

The title compound, $\text{C}_{23}\text{H}_{26}\text{BrNO}_2$, was synthesized by the reaction of 4-bromobenzaldehyde with dimedone and ammonium acetate under microwave irradiation. In the molecule, the dihydropyridine ring adopts a slight boat conformation.

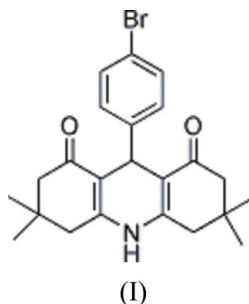
Received 24 November 2005

Accepted 8 December 2005

Online 14 December 2005

Comment

Acridine derivatives, with their interesting chemical and physical properties, have immense utility in the pharmaceutical and dyeing industries, and are well known therapeutic agents (Wysocka-Skrzela & Ledochowski, 1976; Nasim & Brychey, 1979; Thull & Testa, 1994). The discovery of acridines as antimalarial and antitumor agents has attracted the attention of organic chemists and thus led to intensive interest in the synthesis of several drugs based on acridine (Khurana *et al.*, 1990; Matsumoto *et al.*, 1983). We report here the crystal structure of the title compound, (I).



The dihydropyridine ring in (I) is in a slight boat conformation, with atoms N1 and C9 deviating from the C7/C8/C10/C11 mean plane by 0.077 (3) and 0.150 (3) Å, respectively (Fig. 1). Both cyclohexene rings adopt sofa conformations: atom C3 deviates from the C1/C2/C6/C7/C8 by 0.617 (3) Å and atom C13 deviates from the C10/C11/C12/C16/C17 plane by 0.651 (3) Å. The dihedral angle between the C1/C2/C6/C7/C8 and C7/C8/C10/C11 planes is 2.61 (7)° and that between the C7/C8/C10/C11 and C10/C11/C12/C16/C17 planes is 7.55 (7)°. In the crystal structure, molecules are linked *via* N—H...O hydrogen bonds, forming one-dimensional chains in the *a*-axis direction (Table 1 and Fig. 2).

Experimental

The title compound was prepared by the reaction of 4-bromobenzaldehyde (1 mmol) with dimedone (2 mmol) and ammonium acetate (1 mmol) under microwave irradiation (yield 93%; m.p. >574 K). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of (I) of an ethanol solution.

Crystal data

$C_{23}H_{26}BrNO_2$
 $M_r = 428.36$
 Orthorhombic, $Pna2_1$
 $a = 14.1598$ (16) Å
 $b = 14.0629$ (16) Å
 $c = 10.8624$ (12) Å
 $V = 2163.0$ (4) Å³
 $Z = 4$
 $D_x = 1.315$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 2822 reflections
 $\theta = 2.8\text{--}23.0^\circ$
 $\mu = 1.92$ mm⁻¹
 $T = 294$ (2) K
 Block, yellow
 $0.26 \times 0.20 \times 0.16$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.610$, $T_{\max} = 0.736$
 11747 measured reflections

3083 independent reflections
 2016 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.052$
 $\theta_{\text{max}} = 26.4^\circ$
 $h = -17 \rightarrow 17$
 $k = -16 \rightarrow 17$
 $l = -13 \rightarrow 6$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.091$
 $S = 1.00$
 3083 reflections
 252 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0196P)^2 + 1.345P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.42$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.54$ e Å⁻³
 Absolute structure: Flack (1983), 750 Friedel pairs
 Flack parameter: 0.003 (13)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O2^i$	0.87 (3)	1.88 (2)	2.726 (4)	165 (4)

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

H atoms bonded to C atoms were placed in geometrically idealized positions ($C-H = 0.93\text{--}0.98$ Å) and allowed to ride on their parent atoms, with $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$ for methyl H atoms and $1.2U_{\text{eq}}(C)$ for other H atoms. The H atom bonded to N1 was refined isotropically.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b) and PLATON (Spek, 2003); software used to prepare material for publication: SHELXTL.

We thank the Natural Science Foundation of China (No. 20372057), the Key Laboratory of Organic Synthesis of Jiangsu Province, College of Chemistry and Chemical Engineering, Suzhou University Open Foundation (No. JSK011) and the Key Laboratory of Biotechnology for Medicinal Plants of Jiangsu Province (No. 01AXL 14).

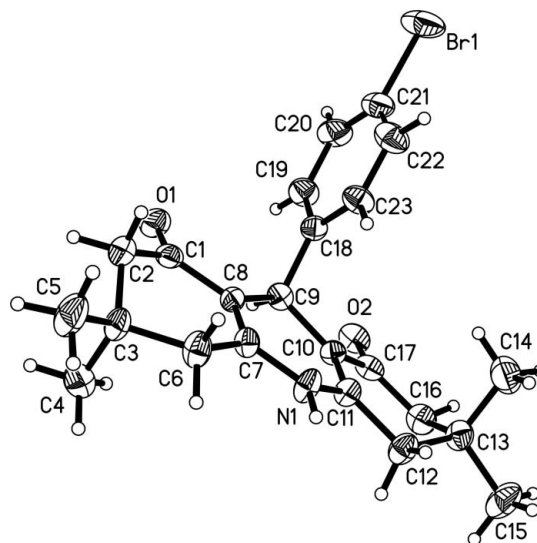


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

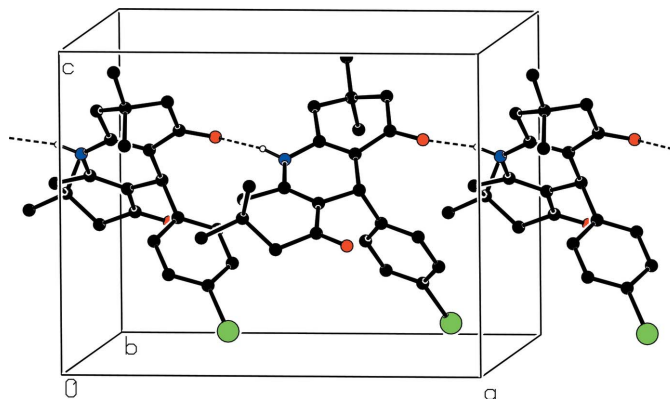


Figure 2

View (Spek, 2003) of the hydrogen-bonded (dashed lines) chain in (I).

References

- Bruker (1998). SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (1999). SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Khurana, J. M., Maikap, G. C. & Mehta, S. (1990). *Synthesis*, pp. 731–732.
 Matsumoto, H., Arai, T., Takahashi, M., Ashizawa, T., Nakano, T. & Nagai, Y. (1983). *Bull. Chem. Soc. Jpn.* **56**, 3009–3014.
 Nasim, A. & Brychey, T. (1979). *Mutat. Res.* **65**, 261–288.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997a). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
 Thull, U. & Testa, B. (1994). *Biochem. Pharmacol.* **47**, 2307–2310.
 Wysocka-Skrzela, B. & Ledochowski, A. (1976). *Rocz. Chem.* **50**, 127–131.