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10-Amino-3,3,6,6-tetramethyl-9-(4-bromophenyl)-3,4,5,6,9,10-hexahydroacridine-1,8(2*H*,7*H*)-dione

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The title compound, $C_{23}H_{27}BrN_2O_2$, has been synthesized by the reaction of 3,3,6,6-tetramethyl-9-(4-bromophenyl)-1,8-dioxo-2,3,4,5,6,7-hexahydroanthene with hydrazine in alcohol under microwave irradiation. X-ray analysis reveals that the dihydropyridine ring adopts a boat conformation.

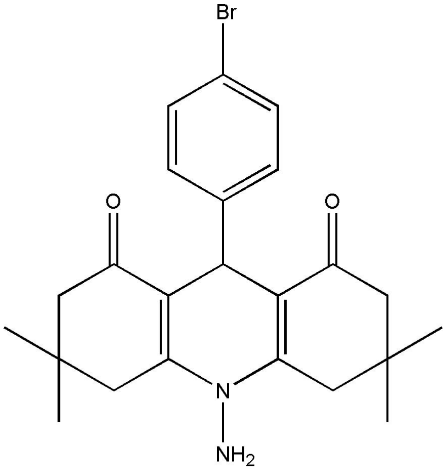
Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.102
Data-to-parameter ratio = 17.8

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

Comment

Acridine belongs to a special class of compounds, which are of interest not only because of their chemical and physical properties, but also due to their immense utility in the pharmaceutical and dye industries. The discovery of acridines as antimalarial and antitumour agents has attracted the attention of organic chemists and thus led to intense interest in the synthesis of several drugs based on acridine (Khurana *et al.*, 1990; Matsumoto *et al.*, 1983; Nakano *et al.*, 1982). Chemical modification of the acridine ring system, such as the introduction of an aryl group on the N atom of acridine, causes laser activity (Murugan *et al.*, 1998). In this paper, we report the crystal structure of the title compound, (I).



(I)

In the molecule of (I), the dihydropyridine ring adopts a boat conformation, with atoms N1 and C3 deviating from the C1/C2/C4/C5 plane by 0.216 (3) and 0.441 (3) Å, respectively (Fig. 1). Both cyclohexene rings adopt envelope conformations: atom C8 deviates from the C1/C2/C6/C7/C9 plane by 0.641 (3) Å and atom C12 deviates from the C4/C5/C13/C11/C10 plane by 0.617 (3) Å. The dihedral angle between the C1/C2/C4/C5 plane and the C14–C19 benzene ring is 85.07 (7)°.

The molecules of (I) are connected *via* N—H···O and C—H···O/Br hydrogen bonds (Table 2), forming a three-dimensional network (Fig. 2).

Experimental

The title compound, (I), was prepared by the reaction of 3,3,6,6-tetramethyl-9-(4-bromophenyl)-1,8-dioxo-2,3,4,5,6,7-hexahydroanthene (1 mmol) with hydrazine (5 mmol) in ethanol (2 ml) under microwave irradiation. Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol (95%) solution (yield 85%; m.p. 553–554 K).

Crystal data

$C_{23}H_{27}BrN_2O_2$
$M_r = 443.38$
Orthorhombic, *Pbca*
$a = 11.8053\ (10)$ Å
$b = 15.6404\ (15)$ Å
$c = 22.432\ (2)$ Å
$V = 4141.8\ (6)$ Å^3
$Z = 8$
$D_x = 1.422$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 17 928 reflections
$\theta = 3.0$–$27.5°$
$\mu = 2.01$ mm^{-1}
$T = 193\ (2)$ K
Chunk, yellow
$0.80 \times 0.60 \times 0.50$ mm

Data collection

Rigaku Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min} = 0.251$, $T_{\max} = 0.367$
43 797 measured reflections
4743 independent reflections
4372 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 27.5°$
$h = -15 \rightarrow 15$
$k = -18 \rightarrow 20$
$l = -23 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.102$
$S = 1.16$
4743 reflections
266 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 3.1966P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.77$ e Å^{-3}
$\Delta\rho_{\min} = -0.84$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C17	1.900 (2)	C2—C6	1.454 (3)
O1—C6	1.235 (3)	C2—C3	1.516 (3)
O2—C10	1.229 (3)	C3—C4	1.499 (3)
N1—C1	1.392 (3)	C3—C14	1.528 (3)
N1—C5	1.394 (3)	C4—C5	1.363 (3)
N1—N2	1.422 (2)	C6—C7	1.513 (3)
C1—C2	1.357 (3)	C7—C8	1.536 (3)
C1—C9	1.505 (3)	C8—C9	1.537 (3)
C1—N1—C5	119.52 (16)	C5—N1—N2	117.00 (16)
C1—N1—N2	121.76 (16)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2$^{\rm i}$	0.90 (3)	2.33 (3)	3.220 (2)	173 (2)
N2—H2*B*···O1$^{\rm ii}$	0.89 (3)	2.27 (3)	3.046 (3)	145 (2)
C20—H20*B*···Br1$^{\rm iii}$	0.98	2.98	3.910 (2)	159
C11—H11*A*···Br1$^{\rm iv}$	0.99	2.84	3.797 (2)	163
C9—H9*A*···O2$^{\rm i}$	0.99	2.38	3.323 (3)	160

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

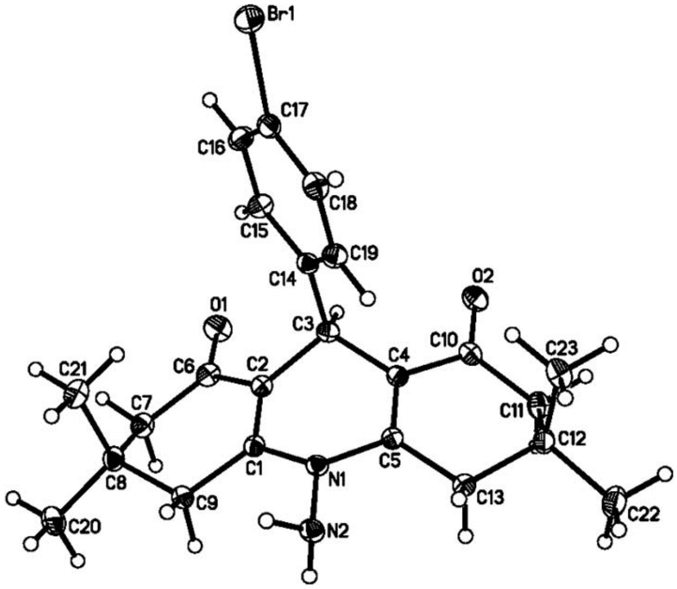


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

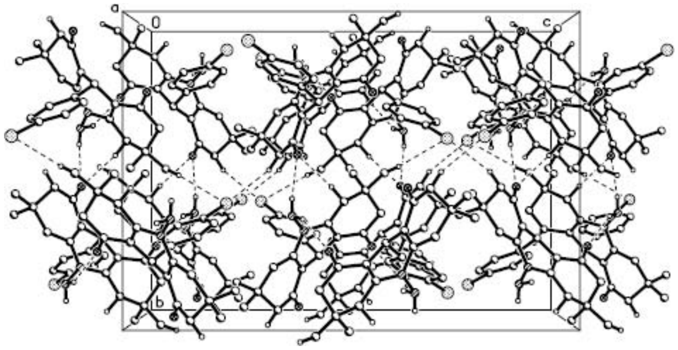


Figure 2
The molecular packing diagram of (I), projected along the *a* axis. Broken lines indicate hydrogen bonds. H atoms have been omitted, except those involved in hydrogen bonding.

The H atoms bonded to the N atom were located in a difference density map and refined isotropically. H atoms bonded to C atoms were located geometrically and treated as riding, with C—H distances in the range 0.95–1.00 Å and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and $1.2U_{\rm eq}({\rm C})$ for others.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2000–2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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