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9-(4-Bromophenyl)-3,3,7-trimethyl-3,4-dihydroacridin-1(2*H*)-one

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.032
wR factor = 0.086
Data-to-parameter ratio = 14.4

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

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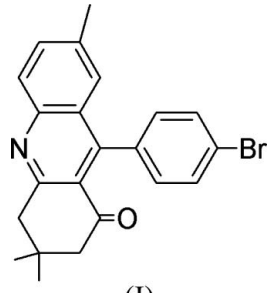
The title compound, $C_{22}H_{20}BrNO$, was synthesized by the reaction of 4-bromobenzaldehyde with *p*-toluidine and 5,5-dimethylcyclohexane-1,3-dione in glycol under microwave irradition. The terminal saturated six-membered ring of the dihydroacridine moiety has a twist-boat conformation. The crystal packing is mainly stabilized by van der Waals forces.

Comment

Acridine and its derivatives inhibit HIV-1 reverse transcriptase by intercalating the template-primer hybrid (Cellai *et al.*, 1994). Such compounds are known as antimicrobial (Al-Ashmawi *et al.*, 1994) and antitumour agents (Wang *et al.*, 1993). They are also used for the treatment of urinary incontinence (Ohnmacht *et al.*, 1993). In a continuation of our structural study of acridinedione derivatives (Guo *et al.*, 2004; Tu *et al.*, 2004), we report here the crystal structure of the title compound, (I) (Fig. 1).



(I)

The bond lengths and angles in (I) are within normal ranges (Xin *et al.*, 1980; Table 1). The six-membered ring C1–C4/C11/C12 has a twist-boat conformation, with atoms C2 and C3 deviating from the C1/C4/C11/C12 mean plane by 0.203 and −0.526 Å, respectively. The pyridine ring makes dihedral angles of 85.67 (8) and 1.36 (15)°, respectively, with the C17–C22 benzene ring and the C1/C4/C11/C12 plane. The crystal packing (Fig. 2) is mainly stabilized by van der Waals forces.

Experimental

The title compound, (I), was prepared by the reaction of 4-bromobenzaldehyde (1 mmol) with *p*-toluidine (1 mmol) and 5,5-dimethylcyclohexane-1,3-dione (1 mmol) in glycol (1 ml) under microwave irradiation (yield 93%; m.p. 513–514 K). Single crystals of (I) suitable for X-ray diffraction were obtained from an ethanol solution by slow evaporation.

Crystal data

$C_{22}H_{20}BrNO$
$M_r = 394.30$
Triclinic, $P\overline{1}$
$a = 10.045$ (2) Å
$b = 10.247$ (2) Å
$c = 11.261$ (2) Å
$\alpha = 111.134$ (2)°
$\beta = 112.428$ (3)°
$\gamma = 99.831$ (3)°
$V = 933.3$ (4) Å^3
$Z = 2$
$D_x = 1.403$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2153 reflections
$\theta = 2.2$–$28.2°$
$\mu = 2.21$ mm^{-1}
$T = 298$ (2) K
Block, light yellow
0.43 × 0.35 × 0.21 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.450$, $T_{max} = 0.654$
4925 measured reflections
3255 independent reflections
2609 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 25.0°$
$h = -6 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.086$
$S = 1.03$
3255 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.1846P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C12	1.320 (3)	C9—C10	1.421 (3)
N1—C13	1.364 (3)	C10—C11	1.390 (3)
C9—C13	1.419 (3)	C11—C12	1.430 (3)
C12—N1—C13	118.2 (2)	C13—C9—C10	117.9 (2)
C1—C2—C3	113.7 (2)	C12—C11—C1	118.8 (2)
C2—C3—C4	107.0 (2)	N1—C12—C11	123.1 (2)
C12—C4—C3	114.6 (2)	N1—C13—C9	123.2 (2)
C1—C2—C3—C14	−61.0 (3)	C10—C11—C12—N1	−0.2 (3)
C1—C2—C3—C4	59.7 (3)	C1—C11—C12—C4	−0.3 (3)
C2—C3—C4—C12	−50.1 (3)	C3—C4—C12—C11	22.4 (3)
C2—C1—C11—C12	9.1 (3)	C12—N1—C13—C9	−1.2 (4)
C13—N1—C12—C11	1.7 (3)	C10—C9—C13—N1	−0.7 (3)

H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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); software used to prepare material for publication: *SHELXTL*.

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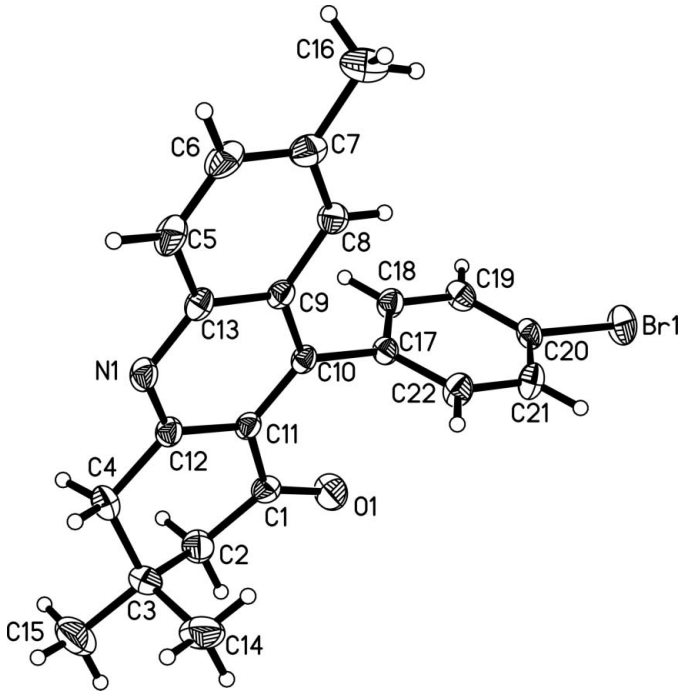

Figure 1
View of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

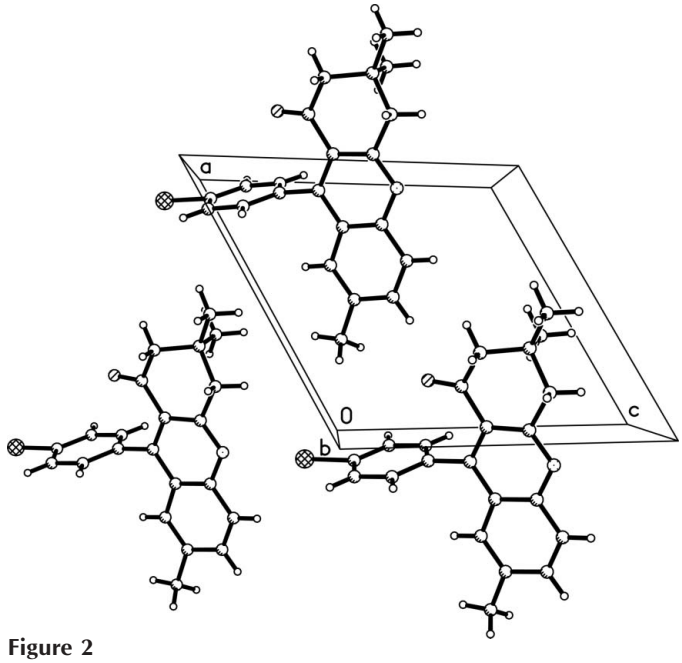

Figure 2
The crystal packing in (I), viewed approximately along the *b* axis.

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