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# 4-(4-Bromophenyl)-3,7,7-trimethyl-4,7,8,9-tetrahydro-2*H*-pyrazolo[3,4-*b*]quinolin-5(6*H*)-one

**Debbie Cannon,[a] Antonio Quesada,[a]† Jairo Quiroga,[b] Diana Mejía,[b] Braulio Insuasty,[b] Rodrigo Abonia,[b] Justo Cobo,[c] Manuel Nogueras,[c] Adolfo Sánchez[c] and John Nicolson Low[d]***

[a]Department of Electronic Engineering and Physics, University of Dundee, Dundee DD1 4HN, Scotland, [b]Grupo de Investigación de Compuestos Heterocíclicos., Departamento de Química, Universidad de Valle, AA 25360 Cali, Colombia, [c]Departamento de Química Inorgánica y Orgánica, Universidad de Jaén, 23071 Jaén, Spain, and [d]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland

† Antonio Quesada is a visiting researcher from the Departamento de Química, Inorgánica y Orgánica, Universidad de Jaén, Spain.

Correspondence e-mail: jnlow111@hotmail.com

**Key indicators**

Single-crystal X-ray study
*T* = 150 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.035
*wR* factor = 0.096
Data-to-parameter ratio = 13.5

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



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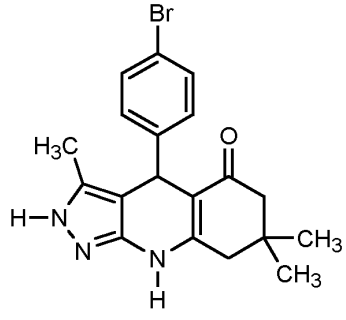
The title compound, $C_{19}H_{20}BrN_3O$, has a supramolecular structure of hydrogen bonding comprising N—H···O bonds which form a series of anti-parallel *C*(8) chains linked together by N—H···N $R_2^2(8)$ base-paired motifs which together form corrugated sheets containing $R_6^6(34)$ rings. This is one of a series of four substituted 3,7,7-trimethyl-4,7,8,9-tetrahydro-2*H*-pyrazolo[3,4-*b*]quinolin-5(6*H*)-one compounds which all have identical supramolecular structures.

## Comment

Pyrazolo[3,4-*b*]quinolines are of interest as possible antiviral agents (Crenshaw *et al.*, 1976, 1978; Smirnoff & Crenshaw, 1977). Some of their derivatives exhibit parasiticidic properties (Bristol–Meyers Co, 1973), and have been studied as potential antimalarial agents (Stein *et al.*, 1970). Some pyrazolo[3,4-*b*]quinolines have shown bactericidal activity (Farghaly *et al.*, 1989), have also been used as vasodilators (Bell & Ackerman, 1990) and evaluated for enzymatic inhibitory activity (Gatta *et al.*, 1991).

In previous reports, (Quiroga, Hormaza *et al.*, 1998; Quiroga, Insuasty *et al.*, 1998), we have reported an efficient and versatile synthesis of novel 4,7,8,9-tetrahydro-pyrimido- and 4,7,8,9-tetrahydropyrazolo[3,4-*b*]quinolin-5-ones from suitable pyrimidine and pyrazole amines to which dimedone and substituted benzaldehyde afford the ring annelation to quinoline.

Selected bond lengths and angles of the title compound, (I), are given in Table 1, while a view of the molecule is given in Fig. 1. The hydrogen bonding pattern comprises anti-parallel *C*(8), [N2—H2···O51$^{i}$], chains linked together by $R_2^2(8)$, [N9—H9···N1$^{ii}$], base-paired motifs (Bernstein *et al.*, 1995). This combination forms a corrugated sheet which contains $R_6^6(34)$ rings. This structure is shown in Fig. 2. The details of the hydrogen bonds are given in Table 2.




(I)

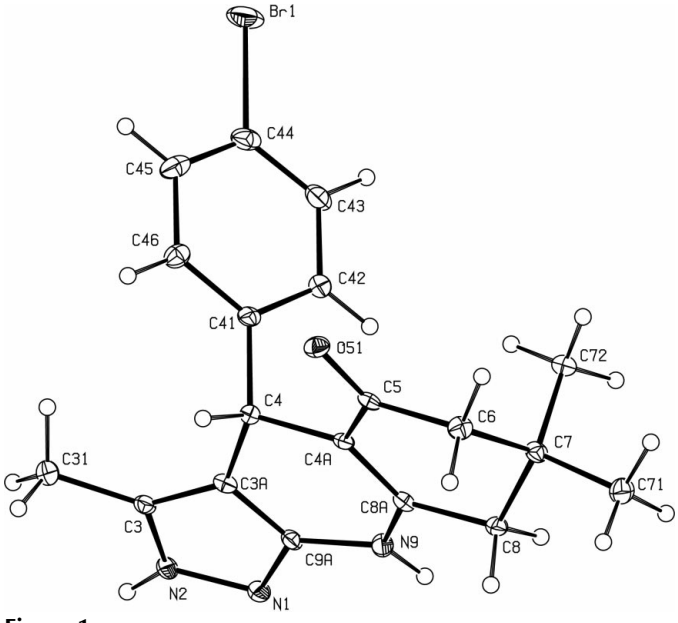



**Figure 1**
A view of the molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Examination of the structure with *PLATON* (Spek, 2000) showed that there were no solvent accessible voids in the crystal lattice.

## Experimental

A solution of 5-aminopyrazole (1 mmol), dimedone (1 mmol) and 4-bromobenzaldehyde (1 mmol) in 15 ml of absolute ethanol were heated to reflux for 20–50 min (thin-layer chromatography control). The reaction mixture was cooled and the solid corresponding to the title compound was filtered off, washed with ethanol, dried and recrystallized from ethanol to afford crystals suitable for diffraction analysis (70% yield, m.p. 587–588 K).

*Crystal data*

$C_{19}H_{20}BrN_3O$
$M_r = 386.29$
Monoclinic, $P2_1/n$
$a = 8.6673\ (2)$ Å
$b = 14.6092\ (3)$ Å
$c = 14.4783\ (5)$ Å
$\beta = 107.287\ (1)^\circ$
$V = 1750.46\ (8)$ Å$^3$
$Z = 4$
$D_x = 1.466$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 4653 reflections
$\theta = 1.0$–$30.5^\circ$
$\mu = 2.36$ mm$^{-1}$
$T = 150\ (1)$ K
Block, colourless
$0.22 \times 0.20 \times 0.13$ mm

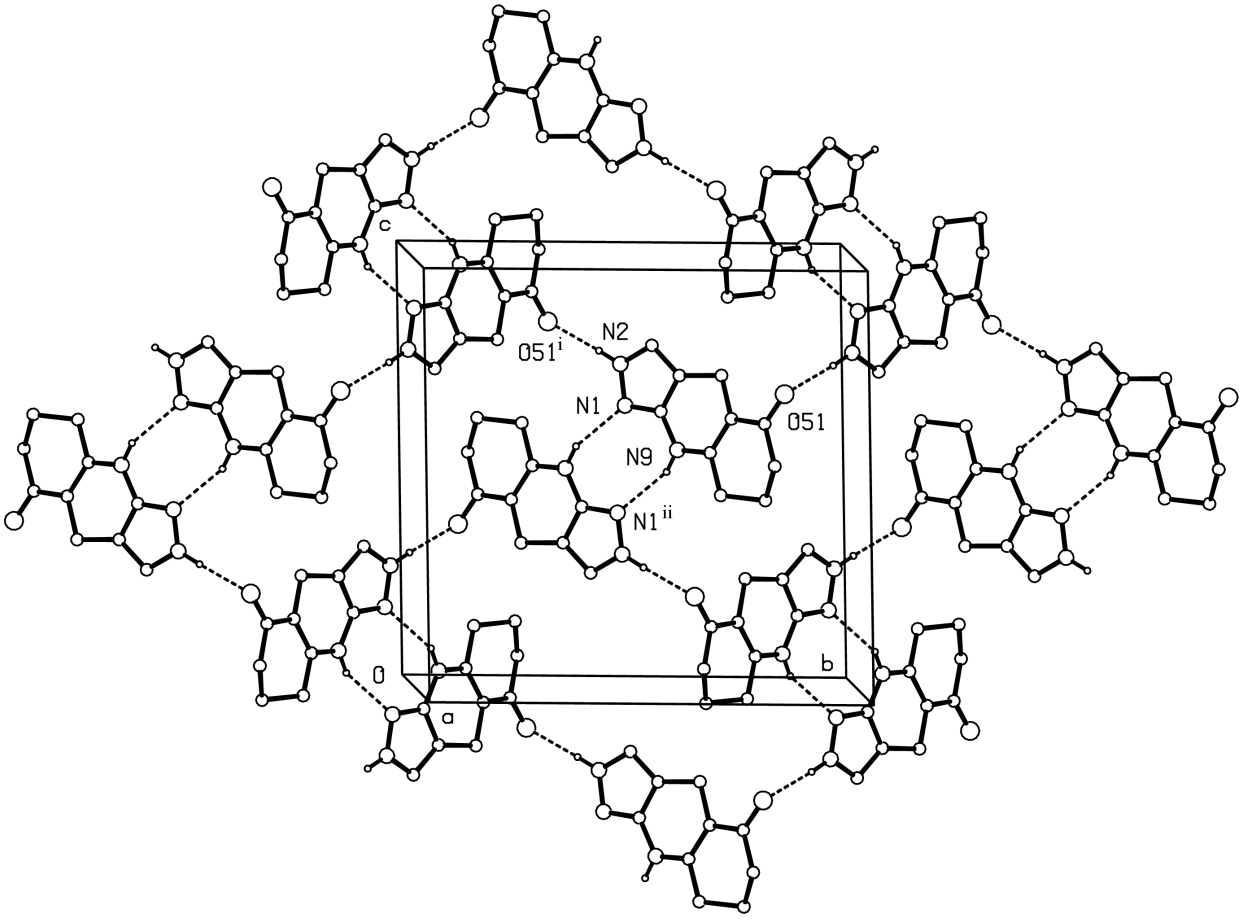



**Figure 2**
View of the hydrogen-bonded sheets lying parallel to [010] showing the $C(8)$ chains, the $R_2^2(8)$ rings and the $R_8^8(34)$ rings. Atom O51$^{i}$ is at $(\frac{1}{2}-x, -\frac{1}{2}+y, \frac{3}{2}-z)$ and atom N1$^{ii}$ is at $(-x, 1-y, 1-z)$.

*Data collection*

| | |
|---|---|
| KappaCCD diffractometer | 2457 reflections with $I > 2\sigma(I)$ |
| $\varphi$ and $\omega$ scans with $\kappa$ offsets | $R_{int} = 0.041$ |
| Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997) | $\theta_{max} = 25.0^\circ$ |
| | $h = -10 \rightarrow 9$ |
| $T_{min} = 0.620$, $T_{max} = 0.757$ | $k = -15 \rightarrow 17$ |
| 10 467 measured reflections | $l = -14 \rightarrow 17$ |
| 2971 independent reflections | Intensity decay: negligible |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 1.0640P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.035$ | |
| $wR(F^2) = 0.096$ | |
| $S = 0.98$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 2971 reflections | $\Delta\rho_{max} = 0.34$ e Å$^{-3}$ |
| 220 parameters | $\Delta\rho_{min} = -0.69$ e Å$^{-3}$ |
| H-atom parameters constrained | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Br1—C44 | 1.901 (3) | N2—C3 | 1.348 (3) |
| N1—C9*A* | 1.329 (3) | C8*A*—N9 | 1.357 (3) |
| N1—N2 | 1.368 (3) | N9—C9*A* | 1.393 (3) |
| | | | |
| C9*A*—N1—N2 | 102.6 (2) | C8*A*—N9—C9*A* | 118.3 (2) |
| C3—N2—N1 | 112.9 (2) | | |

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O51$^{i}$ | 0.88 | 1.96 | 2.824 (3) | 168 |
| N9—H9···N1$^{ii}$ | 0.88 | 2.10 | 2.891 (3) | 150 |

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

H atoms were treated as riding atoms, with C—H = 0.95–1.00 Å and N—H = 0.88 Å.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC, X-ray Crystallographic Service, University of Southampton, using an Enraf–Nonius KappaCCD diffractometer. The authors thank the staff for all their help and advice. We are grateful to the Ministerio de Educación y Cultura for the award of a grant to one of the authors (AQ).

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