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3,7,7-Trimethyl-4-phenyl-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) = 0.003 Å
R factor = 0.056
wR factor = 0.148
Data-to-parameter ratio = 17.4

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

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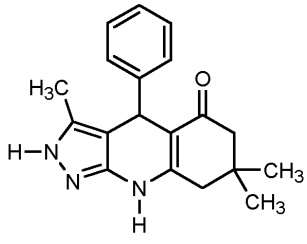
The title compound, $C_{19}H_{21}N_3O$, has a supramolecular structure of hydrogen bonding comprising N—H···O bonds which form a series of anti-parallel *C*(8) chains linked together by N—H···N $R_2^2(8)$ base-paired motifs which together form corrugated sheets containing $R_6^6(34)$ rings. This is one 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.



(I)

Selected bond lengths and angles for the title compound, (I), are given in Table 1 and a view of the molecule is shown 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 shown in Fig. 2. The details of the hydrogen bonds are given in Table 2.

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

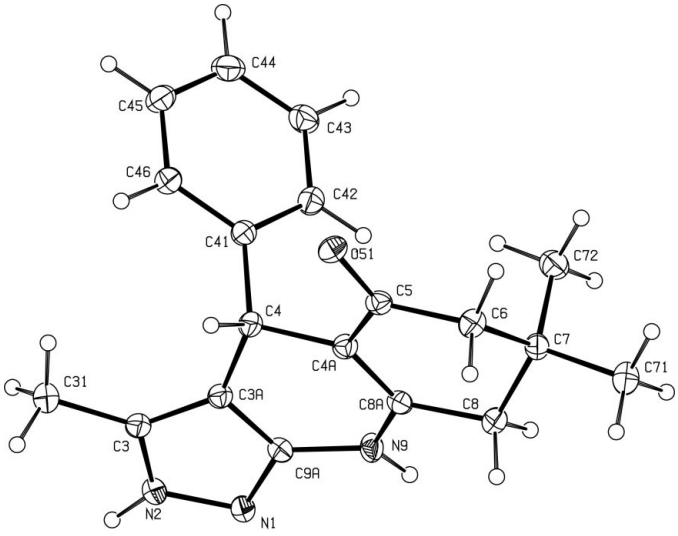


Figure 1
A view of the molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Experimental

A solution of 5-aminopyrazole (1 mmol), dimedone, (1 mmol) and benzaldehyde (1 mmol) in 15 ml of absolute ethanol was 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 suitable crystals for diffraction (60% yield, m.p. 502 K).

Crystal data

$C_{19}H_{21}N_3O$
$M_r = 307.39$
Monoclinic, $P2_1/n$
$a = 10.0870\ (5)$ Å
$b = 14.1978\ (5)$ Å
$c = 11.1928\ (8)$ Å
$\beta = 96.0340\ (14)^\circ$
$V = 1594.08\ (15)$ Å^3
$Z = 4$
$D_x = 1.281$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6601 reflections
$\theta = 2.9$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 150\ (1)$ K
Prism, colourless
$0.16 \times 0.14 \times 0.08$ mm

Data collection

Kappa–CCD diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
$T_{min} = 0.987$, $T_{max} = 0.994$
23 946 measured reflections
3681 independent reflections
1960 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 27.6^\circ$
$h = -13 \rightarrow 13$
$k = -18 \rightarrow 18$
$l = -14 \rightarrow 14$
Intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.148$
$S = 0.94$
3681 reflections
211 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0733P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

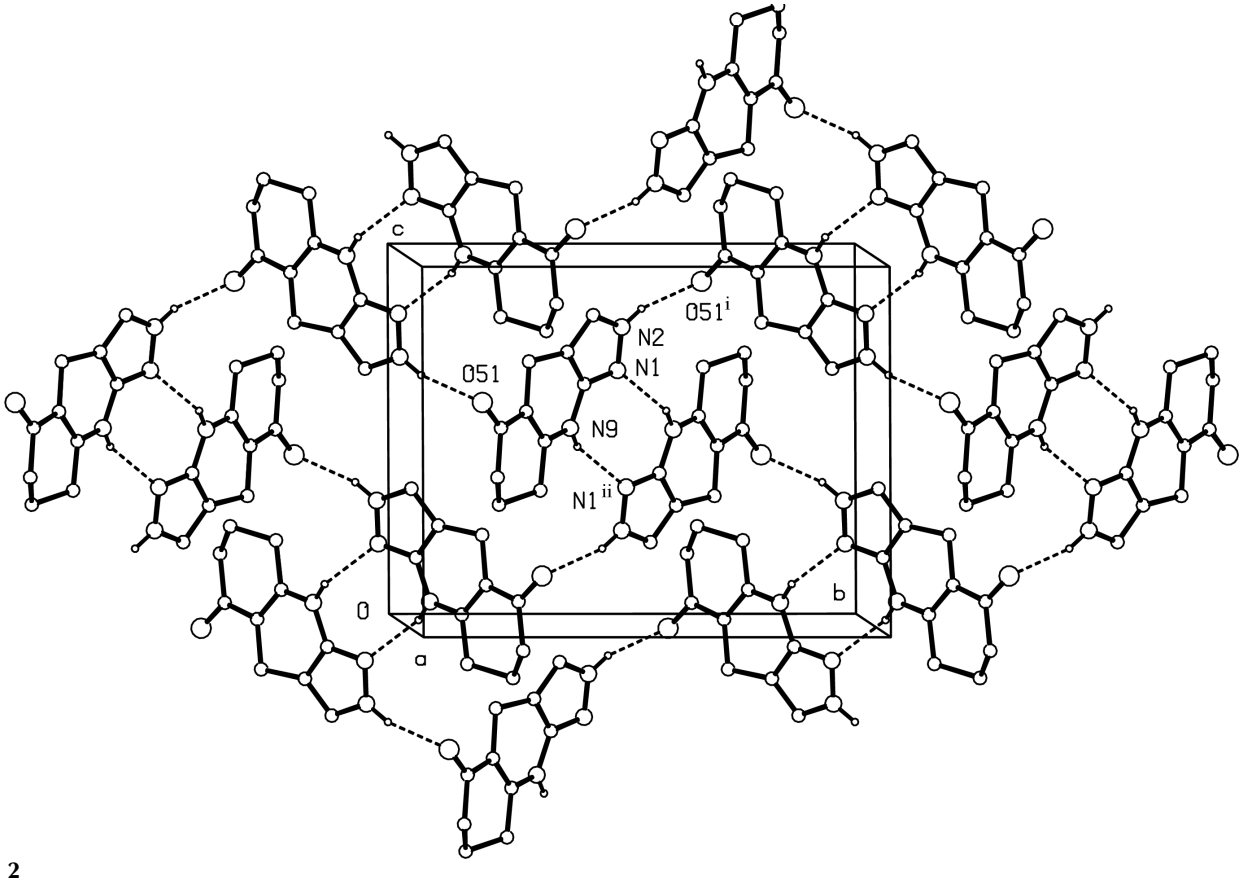


Figure 2
View of the hydrogen bonded sheets lying parallel to [010] showing the $C(8)$ chains, the $R_2^2(8)$ rings and the $R_8^8(34)$ rings. Atom O51^{i} is at $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$ and atom N1^{ii} is at $(-x, 1 - y, 1 - z)$.

Table 1
Selected geometric parameters (Å, °).

N1—C9*A*	1.332 (2)	C8*A*—N9	1.358 (2)
N1—N2	1.366 (2)	N9—C9*A*	1.396 (2)
N2—C3	1.350 (3)		
C9*A*—N1—N2	101.9 (2)	N1—C9*A*—N9	122.9 (2)
C3—N2—N1	113.5 (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	2.03	2.861 (2)	158
N9—H9···N1^{ii}	0.88	2.10	2.885 (2)	148

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