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3,7,7-Trimethyl-4-(β-naphthyl)-4,7,8,9-tetrahydro-2*H*-pyrazolo[3,4-*b*]quinolin-5(6*H*)-one

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.051
wR factor = 0.137
Data-to-parameter ratio = 18.0

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

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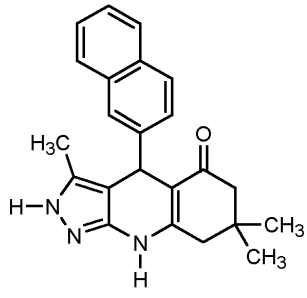
The title compound, $C_{23}H_{23}N_3O$, has a supramolecular structure of hydrogen bonding comprising N—H···O bonds which form a series of anti-parallel *C*(8) chains linked together by N—H···N $R_2^2(8)$ base-paired motifs which together form corrugated sheets containing $R_6^6(34)$ rings. This is one 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 bonds and angles for the title compound, (I), are given in Table 1 and a molecular view 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 is shown in Fig. 2. The details of the hydrogen bonds are given in Table 2.

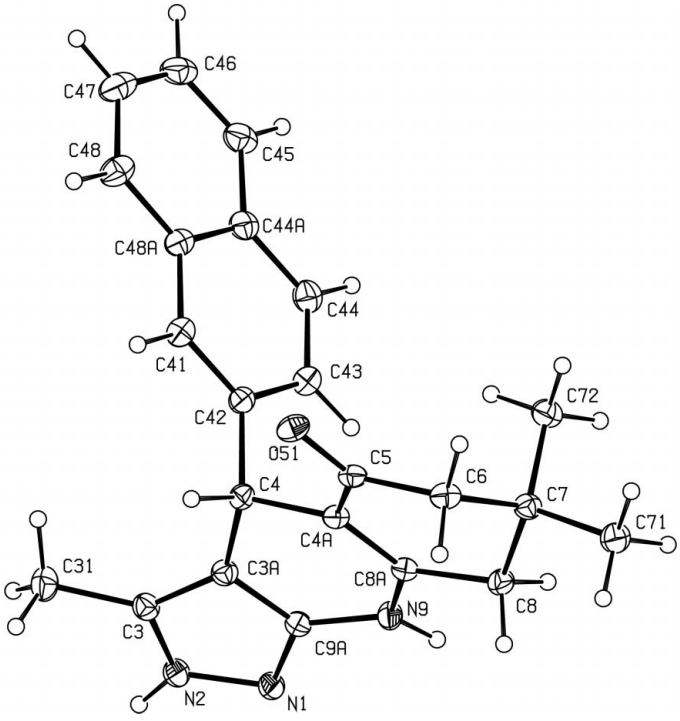


Figure 1
A view of the molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

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 2-naphthaldehyde (1 mmol) in 15 ml of absolute ethanol was heated to reflux for 20–50 min (thin-layer chromatography control control). The reaction mixture was cooled and the solid corresponding to the title compound was filtered out, washed with ethanol, dried and recrystallized from ethanol to afford suitable crystals for diffraction. 65% yield, m.p. 602 K).

Crystal data

$C_{23}H_{23}N_3O$
$M_r = 357.44$
Monoclinic, $P2_1/c$
$a = 9.1222\ (18)$ Å
$b = 15.281\ (3)$ Å
$c = 14.346\ (3)$ Å
$\beta = 106.13\ (3)^\circ$
$V = 1921.0\ (7)$ Å^3
$Z = 4$
$D_x = 1.236$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4401 reflections
$\theta = 2.0$–27.6°
$\mu = 0.08$ mm^{-1}
$T = 150\ (1)$ K
Block, colourless
0.35 × 0.18 × 0.14 mm

Data collection

KappaCCD diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
$T_{min} = 0.974$, $T_{max} = 0.989$
16 205 measured reflections
4401 independent reflections
2994 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 27.6^\circ$
$h = -11 \rightarrow 11$
$k = -19 \rightarrow 19$
$l = -18 \rightarrow 18$
Intensity decay: negligible

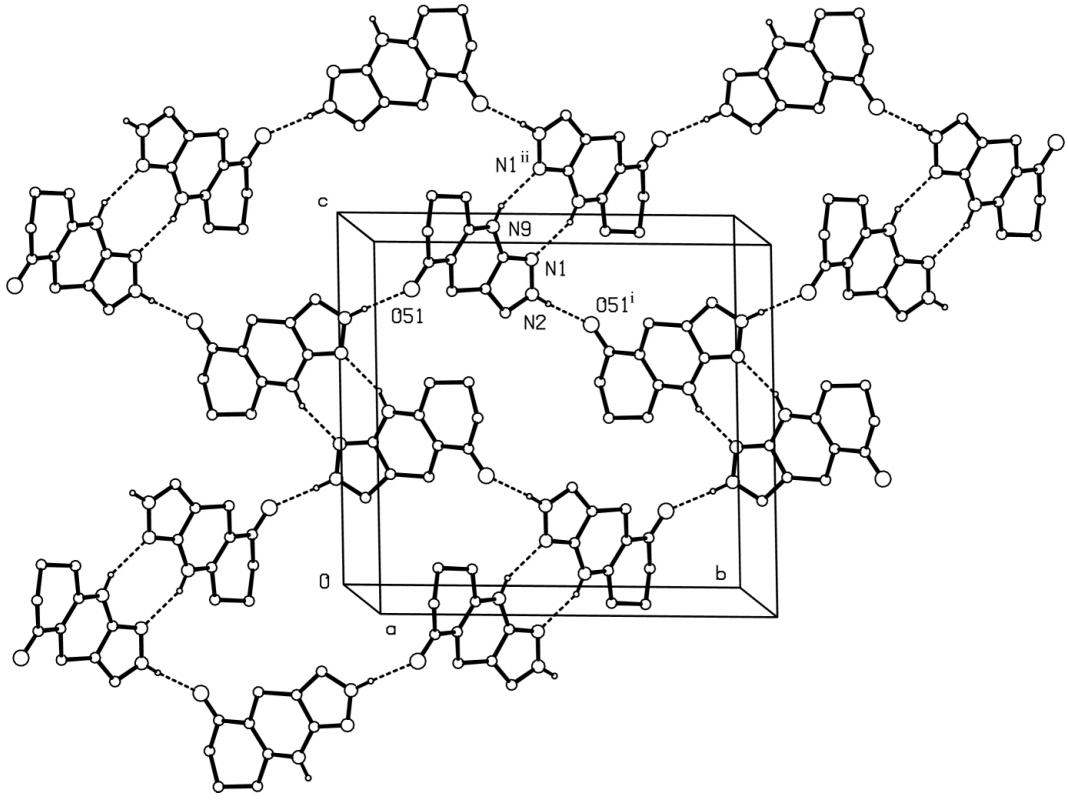


Figure 2
View of the hydrogen-bonded sheets lying parallel to [010] showing the $C(8)$ chains, the $R_2^2(8)$ rings and the $R_8^8(34)$ rings. Atom O51^{i} is at $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$ and atom N1^{ii} is at $(-x, 1 - y, 2 - z)$.

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.051$	$w = 1/[\sigma^2(F_o^2) + (0.0763P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.137$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.03$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
4401 reflections	$\Delta\rho_{min} = -0.28$ e Å^{-3}
245 parameters	

Table 1
Selected geometric parameters (Å, °).

N1—C9*A*	1.328 (2)	C8*A*—N9	1.358 (2)
N1—N2	1.366 (2)	N9—C9*A*	1.390 (2)
N2—C3	1.347 (2)		
C9*A*—N1—N2	102.25 (12)	C8*A*—N9—C9*A*	117.82 (12)
C3—N2—N1	113.42 (12)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O51^{i}	0.88	1.95	2.810 (2)	165
N9—H9···N1^{ii}	0.88	2.05	2.878 (2)	155

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

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 diffractomenter. 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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