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4-(4-Methoxyphenyl)-3,7,7-trimethyl-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
Disorder in main residue
R factor = 0.053
wR factor = 0.141
Data-to-parameter ratio = 13.7

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

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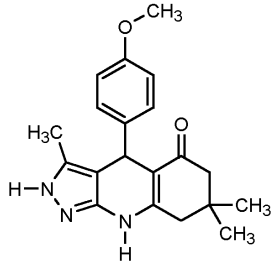
The title compound, $C_{20}H_{23}N_3O_2$, has a supramolecular structure of hydrogen bonding comprising N—H···O bonds which form a series of anti-parallel *C*(8), chains linked together by N—H···N $R_2^2(8)$ base-paired motifs which together form corrugated sheets containing $R_6^6(34)$ rings. This is 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 for the title compound, (I), are given in Table 1 and a view of the molecule is shown in Fig. 1.



(I)

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.

The 4-methoxyphenyl group was disordered on two sites, with occupancies of 0.65 and 0.35. The group is flipped

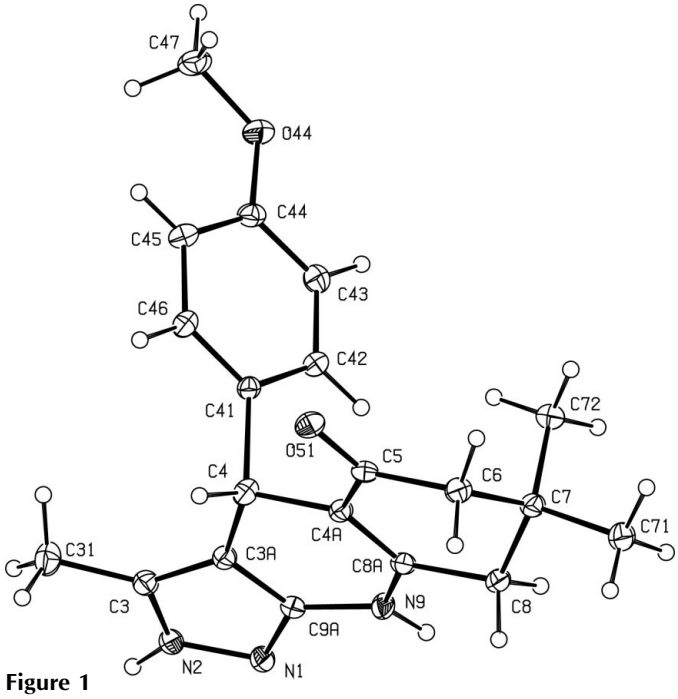


Figure 1
A view of the molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Only the most populated disordered part of the molecule is included.

through nearly 180° about an axis which runs close to the line passing between C4 and C420 and between the C47 and C427 methoxy atoms, the second of each atom pair being in the minor component.

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-methoxybenzaldehyde (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 (61% yield, m.p. 570 K).

Crystal data

$C_{20}H_{23}N_3O_2$
M_r = 337.41
Monoclinic, $P2_1/n$
a = 8.5820 (3) Å
b = 14.7031 (5) Å
c = 14.5369 (8) Å
β = 106.0610 (15)°
V = 1762.70 (13) Å^3
Z = 4
D_x = 1.271 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3956 reflections
θ = 2.0–27.5°
μ = 0.08 mm^{-1}
T = 150 (1) K
Prism, colourless
0.38 × 0.24 × 0.18 mm

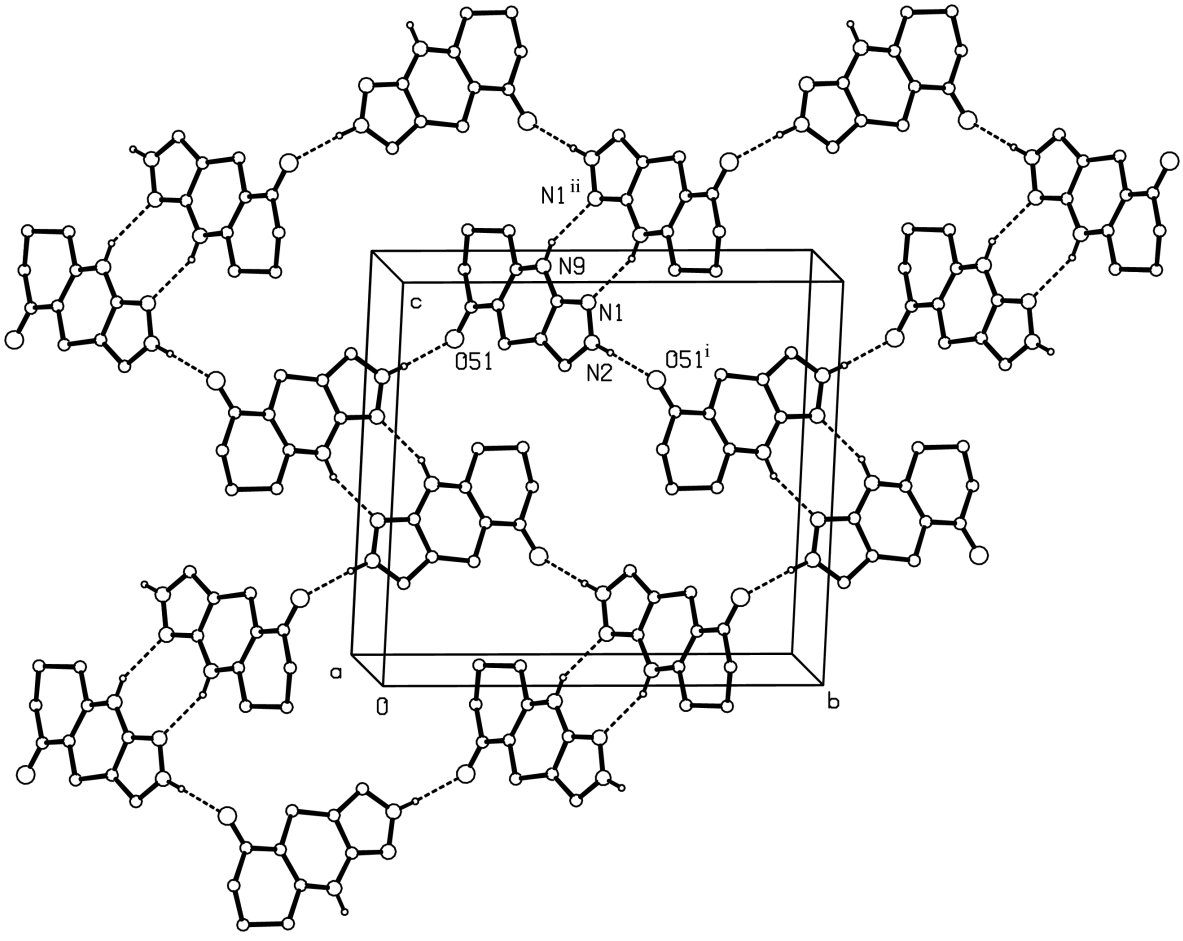


Figure 2
View of the hydrogen bonded sheets lying parallel to [010] showing the $C(8)$ chains, the $R_2^2(8)$ rings and the $R_8^8(34)$ rings. Atom O51^{i} is at ($\frac{3}{2} - x$, $\frac{1}{2} + y$, $\frac{3}{2} - z$) and atom N1^{ii} is at ($2 - x$, $1 - y$, $2 - z$).

Data collection

KappaCCD diffractometer	2662 reflections with $(I) > 2\sigma(I)$
φ and ω scans with κ offsets	$R_{int} = 0.029$
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)	$\theta_{max} = 27.5^\circ$
$T_{min} = 0.969$, $T_{max} = 0.985$	$h = -11 \rightarrow 10$
11 320 measured reflections	$k = -18 \rightarrow 16$
3956 independent reflections	$l = -15 \rightarrow 18$
	Intensity decay: negligible

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.4575P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.053$	$(\Delta/\sigma)_{max} = 0.002$
$wR(F^2) = 0.141$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
$S = 1.03$	$\Delta\rho_{min} = -0.30$ e Å^{-3}
3956 reflections	
289 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—C9*A*	1.335 (2)	C8*A*—N9	1.357 (2)
N1—N2	1.358 (2)	C9*A*—N9	1.386 (2)
N2—C3	1.345 (2)		
C9*A*—N1—N2	102.49 (14)	C8*A*—N9—C9*A*	118.34 (15)
C3—N2—N1	113.55 (14)		

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 (2)	167
N9—H9···N1^{ii}	0.88	2.07	2.880 (2)	153

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

H atoms were treated as riding atoms with C—H = 0.95–1.00 Å and N—H = 0.88 Å. In the disordered groups, each of the atoms in the pairs C4/C420, C44/C424 and O44/O424 were given equal ADPs. The C4—C41 and C420—C421 bonds were restrained to 1.520 (5) Å. All other parameters in the groups were allowed to refine freely. Although there are some variations from normal phenyl bonds in the minor component, these lie within 3σ of the expected values so no further action was taken.

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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