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11-(4-Chlorophenyl)-10-methyl-8-phenyl-6,8-dihydro-5*H*-benzo[*f*]pyrazolo[3,4-*b*]-quinoline

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.110
Data-to-parameter ratio = 17.3

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

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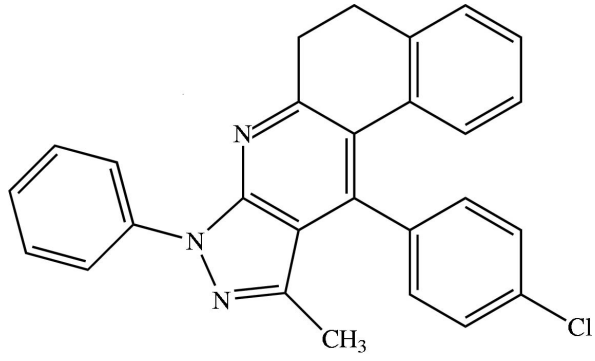
Molecules of the title compound, $C_{27}H_{20}ClN_3$, are linked by two independent C—H···π(arene) hydrogen bonds into chains of edge-fused rings.

Comment

Pyrazolo[3,4-*b*]quinolines are of interest as possible antiviral and antimalarial agents, and because of their other biological properties, such as parasiticidic, bactericidal, vasodilator and enzyme-inhibitory activity (Quiroga *et al.*, 2001). We have recently focused on the synthesis of fused heterocyclic systems containing the pyrazolo[3,4-*b*]quinoline moiety using multi-component cyclocondensation reactions under solvent-free conditions. We describe here the molecular and supramolecular structure of the title compound, (I), prepared using a three-component cyclocondensation involving 5-amino-3-methyl-1-phenylpyrazole, 2-tetralone and 4-chlorobenzaldehyde under solvent-free microwave irradiation.



(I)

Within the pyridine-type ring, the C—N bond lengths (Table 1) are very close to the mean value of 1.337 Å for bonds of this type (Allen *et al.*, 1987), and there is very strong bond fixation in the five-membered ring. The pyridine ring and the benzene ring containing atom C1 are not coplanar, and their planes make a dihedral angle of 25.5 (2)°. The carbocylic ring containing atoms C5 and C6 accordingly adopts a screw-boat conformation (Evans & Boeyens, 1989), with total puckering amplitude Q = 0.537 (2) Å, and ring-puckering parameters θ = 70.5 (2)° and φ = 92.5 (2)° (Cremer & Pople, 1975); the idealized values of the angular parameters for a screw-boat conformer are θ = 67.5° and φ = (60k + 30)°. The dihedral angle between the pyrazole-type ring and aryl ring C81–C86 is 28.1 (2)°, whereas that between the pyridine-type ring and aryl ring C111–C116 is 70.1 (2)°, possibly as a consequence of repulsive interactions between the H atoms bonded to C112 and C116 and those bonded to C101 and C1, respectively.

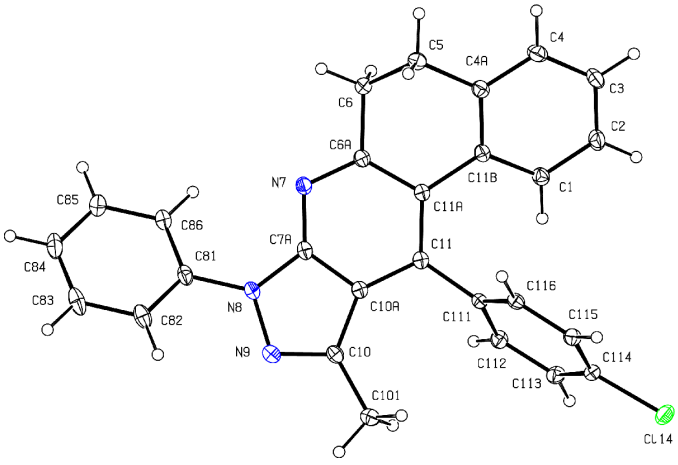


Figure 1
The molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

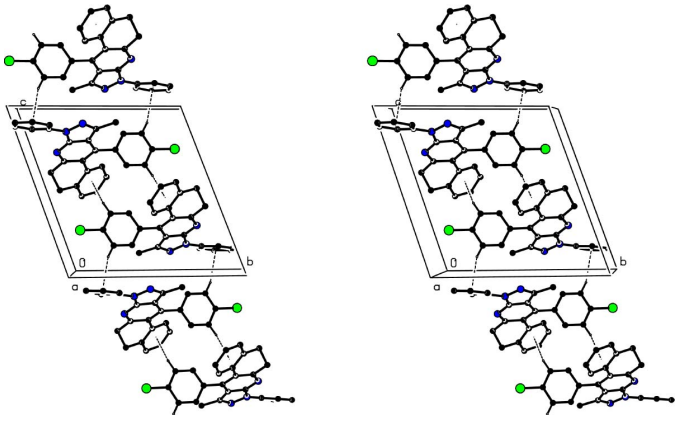


Figure 2
Stereoview of part of the crystal structure of compound (I), showing the formation of a chain of edge-fused rings along [101]. For the sake of clarity, the H atoms not involved in these motifs have been omitted.

The molecules of (I) are linked by two independent C—H···π(arene) hydrogen bonds into a chain of edge-fused rings. Aryl atom C113 in the molecule at (x, y, z) acts as donor to the phenyl ring C81–C86 in the molecule at ($-x$, $1-y$, $-z$), so forming a centrosymmetric ring, centred at $(0, \frac{1}{2}, 0)$. In a similar way, atom C115 at (x, y, z) acts as donor to the fused aryl ring, containing C1, in the molecule at ($1-x$, $1-y$, $1-z$), so generating a second centrosymmetric ring, centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. Propagation by inversion of these two interactions then generates a chain of edge-fused centrosymmetric rings running parallel to the [101] direction (Fig. 2). There are no direction-specific interactions between adjacent chains: C—H···N and C—H···Cl hydrogen bonds, and aromatic π–π stacking interactions are all absent from the structure of (I).

Experimental

Equimolar amounts of 5-amino-3-methyl-1-phenylpyrazole (173 mg, 1.0 mmol), 2-tetralone (146 mg, 1.0 mmol) and 4-chlorobenzaldehyde (140.6 mg, 1.0 mmol) were placed in open Pyrex glass vessels and irradiated in a domestic microwave oven for 4 min at 600 W. The reaction mixture was then extracted with ethanol and, after removal of the solvent, the product was recrystallized from ethanol/dimethylformamide to give crystals suitable for single-crystal X-ray diffraction. Pale-green crystals (m.p. 467 K, yield 58%). MS: (30 eV) m/z (%) 279 (100, M^+), 264 (27).

Crystal data

$C_{27}H_{20}ClN_3$
$M_r = 421.91$
Triclinic, $P\overline{1}$
$a = 7.1270$ (1) Å
$b = 12.6300$ (4) Å
$c = 13.2847$ (4) Å
$\alpha = 107.3380$ (13)°
$\beta = 103.6230$ (17)°
$\gamma = 101.4230$ (18)°
$V = 1061.98$ (5) Å^3
$Z = 2$
$D_x = 1.319$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4871 reflections
$\theta = 3.0$–27.6°
$\mu = 0.20$ mm^{-1}
$T = 120$ (2) K
Plate, pale green
0.53 × 0.20 × 0.08 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.906$, $T_{max} = 0.984$
21 625 measured reflections
4871 independent reflections
3853 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.6°$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.110$
$S = 1.08$
4871 reflections
281 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.2795P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

Table 1
Selected bond lengths (Å).

C6*A*—N7	1.3336 (18)	C10—C10*A*	1.436 (2)
N7—C7*A*	1.3415 (19)	C10*A*—C11	1.4055 (19)
C7*A*—N8	1.3730 (17)	C11—C11*A*	1.403 (2)
N8—N9	1.3791 (17)	C11*A*—C6*A*	1.4324 (19)
N9—C10	1.3190 (19)	C7*A*—C10*A*	1.398 (2)

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

*Cg*1 is the centroid of ring C81–C86, and *Cg*2 is the centroid of ring C1–C4/C4*A*/C11*B*.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C113—H113···*Cg*1^{i}	0.95	2.65	3.5214 (16)	152
C115—H115···*Cg*2^{ii}	0.95	2.90	3.6403 (17)	136

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1-x, 1-y, 1-z$.

All H atoms were located in difference maps in fully ordered sites; they were then treated as riding atoms, with C—H distances of 0.95 (aromatic), 0.98 (methyl) or 0.99 Å (CH$_2$), and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for the methyl group.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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