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1-(*p*-Chlorophenyl)-3,4-dimethyl-5,6,7,8-tetrahydropyrazolo[3,4-*b*]quinoline

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.051
wR factor = 0.128
Data-to-parameter ratio = 15.6

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

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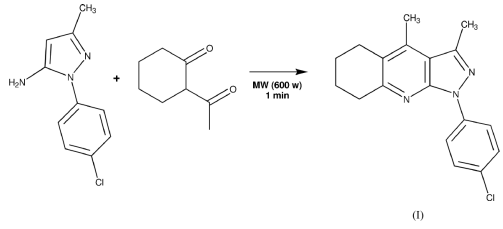
The title compound, $C_{18}H_{18}ClN_3$, shows no unusual features. There is no hydrogen-bonding or π–π stacking.

Comment

In our ongoing studies on the application of free solvent cyclocondensation procedures under microwave irradiation, we have now prepared a pyrazolo[3,4-*b*]quinoline, (I), starting from 5-aminopyrazole and 2-acetylcyclohexanone. There are no unusual bonds or angles in this compound nor are there any intermolecular contacts less than 3.5 Å. The molecule is disordered (Fig. 1).



(I)

The disorder is in the rings defined by C4*A*—C5—C6—C7—C8—C8*A* (major component) and C4*A*—C5—C6*A*—C7*A*—C8—C8*A* (minor component). The nature of the disorder can be seen by examining the displacements of atoms C6 [−0.394 (9) Å] and C7 [0.369 (9) Å], and C6*A* [0.43 (2) Å] and C7A [−0.33 (2) Å] from the mean plane formed by atoms C4*A*/C5/C8/C8*A*. Thus, for the corresponding atom pairs C6/C6*A* and C7/C7*A*, the displacements from the mean plane are similar but opposite in sign.

Experimental

A mixture of 5-amino-3-methyl-1-(4-chlorophenyl)pyrazole (10 mmol) and 2-acetylcyclohexanone (11 mmol) was poured into an open Pyrex-glass vessel and irradiated (600 W) in a domestic microwave oven for 1 min. The solid was collected by filtration, washed with ethanol, dried and recrystallized from DMF–water. The resulting crystals were suitable for X-ray diffraction. M.p. 420 K, yield 64%. Analysis calculated for $C_{18}H_{18}ClN_3$: C 69.34, H 5.82, N 13.48%; found: C 69.28, H 5.78, N 13.55%.

Crystal data

$C_{18}H_{18}ClN_3$
M_r = 311.80
Monoclinic, $P2_1/c$
a = 10.2097 (6) Å
b = 7.5773 (3) Å
c = 20.6921 (12) Å
β = 110.203 (3)°
V = 1502.29 (14) Å^3
Z = 4
D_x = 1.379 Mg m^{-3}
Mo Kα radiation
Cell parameters from 3431 reflections
θ = 3.4–27.6°
μ = 0.25 mm^{-1}
T = 120.0 (2) K
Plate, colourless
0.32 × 0.12 × 0.08 mm

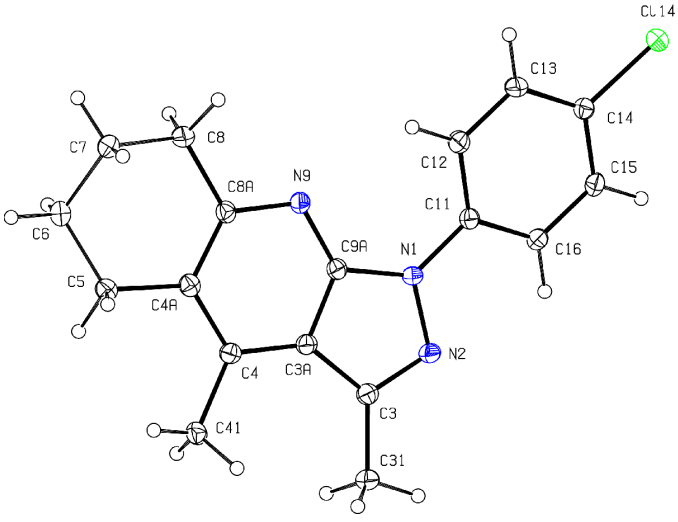


Figure 1
A view of the major component of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Data collection

Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.947, T_{max} = 0.987
22681 measured reflections
3431 independent reflections
2068 reflections with $I > 2\sigma(I)$
R_{int} = 0.097
θ_{max} = 27.6°
$h = -13 \rightarrow 13$
$k = -9 \rightarrow 9$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.128
S = 0.97
3431 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0682P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

H atoms were treated as riding atoms, with aromatic C—H = 0.95 Å, methyl C—H = 0.98 Å and CH_2 C—H = 0.99 Å. The disordered atoms C6/C7 and C6*A*/C7*A* and their attached H atoms, as well as the disordered H atoms attached to C5 and C8, were refined (*SHELXL*97; Sheldrick, 1997) and with a free variable defining the site-occupancy factor. This free variable was allowed to refine and gave values of 0.734 (15) and 0.266 (15) for the occupancies of the major and minor components, respectively. Atoms C6, C7, C6*A* and C7*A* were refined anisotropically.

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, 2003); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

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