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N-[4-(4-Chlorophenyl)-3-methyl-6-oxo-1-phenyl-4,5,6,7-tetrahydro-1*H*-pyrazolo[3,4-*b*]pyridin-5-yl]benzamide

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.051
wR factor = 0.147
Data-to-parameter ratio = 13.6

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

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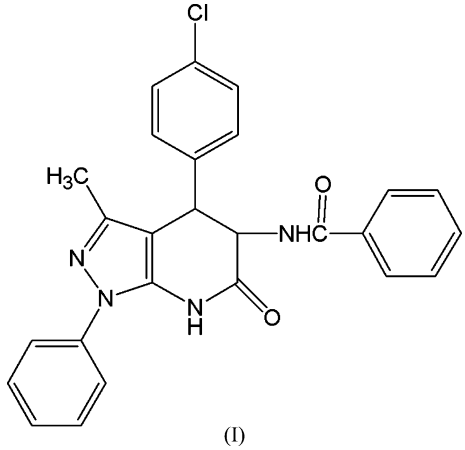
The title compound, $C_{26}H_{21}ClN_4O_2$, has been synthesized by the reaction of 4-(4-chlorobenzylidene)-2-phenyl-1,3-oxazol-5(4*H*)-one with 3-methyl-1-phenyl-1*H*-pyrazol-5-amine in glycol under microwave irradiation. All atoms of the pyrazolo[3,4-*b*]pyridine system, with the exception of two adjacent C atoms carrying the benzamido and oxo substituents (positions 5 and 6), lie in one plane; the displacements of the latter C atoms from this plane are 0.284 (5) and 0.847 (5) Å respectively. Only one of the two NH groups, *viz*. that in the dihydropyridinone ring, participates in the intermolecular hydrogen bonds which link the molecules into infinite chains running along the *b* axis

Comment

1*H*-Pyrazolo[3,4-*b*]pyridine is known to be of significant biological and medicinal importance (Ali *et al.*, 2003; Barreiro *et al.*, 2003). It has been reported that pyrazolo[3,4-*b*]pyridines are potential specific antagonists of nucleic acid metabolism. Derivatives of this heterocyclic ring system have been shown to be substrate inhibitors of purine-requiring enzymes and also exhibit potential non-sedative anxiolytic activity (Bare *et al.*, 1989).



(I)

In this paper, we report the crystal structure of the title compound, (I). Seven atoms of the pyrazolopyridine system (N1, N2, N3, C3, C4, C5 and C6) are coplanar [the maximum deviation from the least-squares plane being 0.012 (3) Å for atom C4], whereas the remaining two atoms of the bicyclic system, C1 and C2, are displaced from this plane by 0.284 (5) and 0.847 (5) Å, respectively.

Although molecule (I) contains two 'active' H atoms (H1 and H4), only one of them, atom H1 of the dihydropyridinone NH group, participates in intermolecular hydrogen bonding

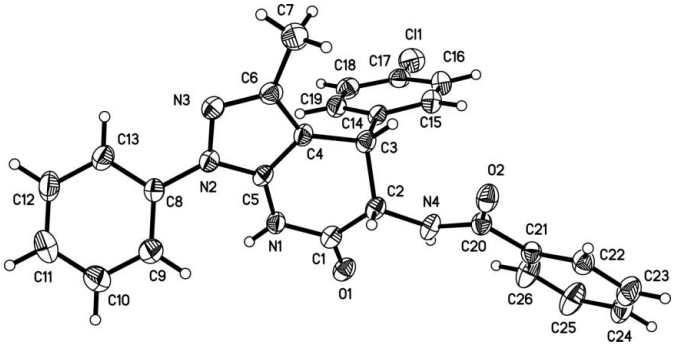


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids.

(see Table 2). These hydrogen bonds link the molecules into infinite chains running along the *b* axis.

Experimental

All reactions were performed in a monomodal EmrysTM Creator, a synthesator from Personal Chemistry (Uppsala, Sweden). In a 10 ml EmrysTM reaction vial, 4-(4-chlorobenzylidene)-2-phenyl-1,3-oxazol-5(4*H*)-one (1 mmol), 3-methyl-1-phenyl-1*H*-pyrazol-5-amine (1 mmol) and glycol (3 ml) were mixed and the vial was capped. The mixture was irradiated for 4 min at 180 W power and 423 K. Upon completion of the reaction, monitored by thin-layer chromatography, the reaction mixture was cooled to room temperature and then poured into cold water. The solid product was filtered off, washed with water and EtOH (95%), and subsequently dried and recrystallized from EtOH (95%) to give the pure product. Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a 95% aqueous ethanol solution (yield 94%; m.p. 528 K).

Crystal data

$C_{26}H_{21}ClN_4O_2$
M_r = 456.92
Monoclinic, $P2_1/n$
a = 9.785 (5) Å
b = 9.658 (5) Å
c = 24.932 (12) Å
β = 101.283 (8)°
V = 2310.7 (19) Å^3
Z = 4
D_x = 1.313 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 298 (2) K
Block, colorless
0.42 × 0.12 × 0.09 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.922, T_{max} = 0.983
11794 measured reflections
4061 independent reflections
1819 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.147
S = 1.00
4061 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.6448P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

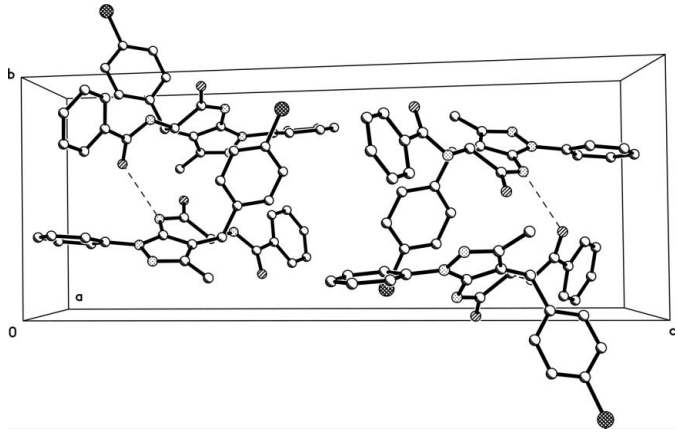


Figure 2
Packing diagram of (I), viewed along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms have been omitted.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.16	2.834 (4)	135

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

All H atoms were positioned geometrically and refined as riding, with N—H = 0.86 Å, C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2 (1.5 for methyl and NH atoms) times U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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References

Ali, A., Taylor, G. E., Ellsworth, K., Harris, G., Painter, R., Silver, L. L. & Young, K. (2003). *J. Med. Chem.* **46**, 1824–1830.
Bare, T. M., McLaren, C. D., Campbell, J. B., Firor, J. W., Resch, J. F., Walters, C. P., Salama, A. I., Meiners, B. A. & Patel, J. B. (1989). *J. Med. Chem.* **32**, 2561–2573.
Barreiro, E. J., Camara, C. A., Verli, H., Brazil-Mas, L., Castro, N. G., Cintra, W. M., Aracava, Y., Rodrigues, C. R. & Fraga, C. A. M. (2003). *J. Med. Chem.* **46**, 1144–1152.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.