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3-Methyl-4-(4-nitrophenyl)-6-oxo-1-phenyl-4,5-dihydropyrazolo[3,4-*b*]pyridine

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The title compound, $C_{19}H_{16}N_4O_3$, was synthesized by the reaction of 5-amino-3-methyl-1-phenylpyrazole with 4-nitrobenzaldehyde and Medrum's acid in glycol under microwave irradiation. X-ray crystal structure analysis reveals that the dihydropyridine ring adopts a distorted half-chair conformation. In the crystal structure, the molecules exist as $N—H\cdots O$ hydrogen-bonded dimers.

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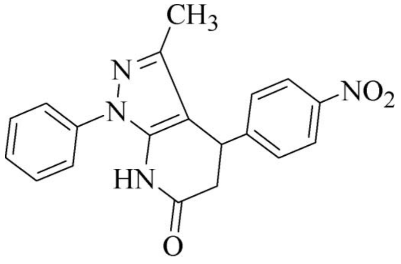
Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.062
wR factor = 0.154
Data-to-parameter ratio = 16.0

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

Comment

The dihydropyrazolo[3,4-*b*]pyridine system has many interesting biological and pharmacological properties, such as vasodilating and antihypertension activities, and also produces a prophylactic effect as a calcium antagonist in stroke-prone spontaneously hypertensive symptoms (Sekikawa *et al.*, 1973; Kuczynski *et al.*, 1979; El-Dean *et al.*, 1991). As part of our program aimed at employing microwave irradiation for the preparation of heterocyclic compounds (Tu *et al.*, 2004), we have recently synthesized dihydropyrazolo[3,4-*b*]pyridine derivatives under microwave irradiation. We report here the crystal structure of the title compound, (I).



(I)

The dihydropyridine ring adopts a distorted half-chair conformation (Fig. 1). The dihedral angle between the planes of the pyrazole ring and the attached phenyl ring is 50.0 (1)°.

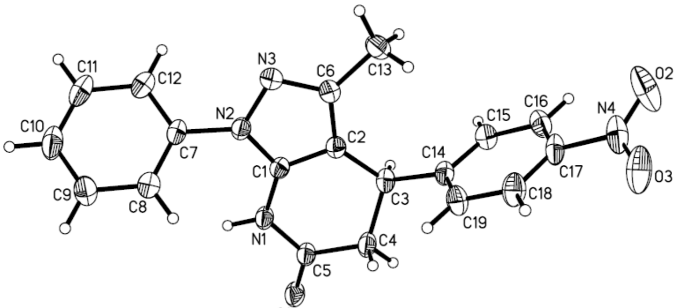


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

The crystal packing shows that the molecules exist as N—H···O hydrogen-bonded dimers (Table 1). The molecular packing is further stabilized by C—H···O interactions (Fig. 2).

Experimental

Compound (I) was prepared by the reaction of 5-amino-3-methyl-1-phenylpyrazole (2 mmol) with 4-nitrobenzaldehyde (2 mmol) and Medrum's acid (2 mmol) in glycol (1 ml) under microwave irradiation for 6 min (yield 80%, m.p. 490–491 K). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution (95%).

Crystal data

$C_{19}H_{16}N_4O_3$
$M_r = 348.36$
Monoclinic, $P2_1/c$
$a = 7.846$ (5) Å
$b = 16.758$ (14) Å
$c = 12.940$ (15) Å
$\beta = 98.374$ (12)°
$V = 1683$ (3) Å^3
$Z = 4$

$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2111 reflections
$\theta = 3.1$–27.5°
$\mu = 0.10$ mm^{-1}
$T = 193$ (2) K
Block, yellow
0.39 × 0.30 × 0.28 mm

Data collection

Rigaku Mercury CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min} = 0.964$, $T_{\max} = 0.974$
18328 measured reflections

3850 independent reflections
3234 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -21 \rightarrow 21$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.154$
$S = 1.13$
3850 reflections
240 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.765P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O3$^{\rm i}$	0.99	2.59	3.465 (5)	148
C12—H12···O3$^{\rm ii}$	0.95	2.58	3.285 (4)	131
C16—H16···O2$^{\rm iii}$	0.95	2.53	3.437 (3)	160
N1—H1···O1$^{\rm iv}$	0.94 (2)	1.87 (2)	2.814 (3)	177 (2)

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $x, y, z-1$; (iii) $-x, -y+1, -z+2$; (iv) $-x+2, -y+1, -z+1$.

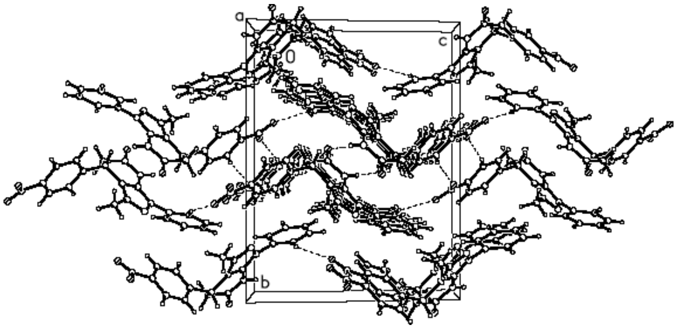


Figure 2
The molecular packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

The H atom of the NH group was located in a difference Fourier map and was refined isotropically. All other H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å, and with $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C) for methyl H atoms and $1.2U_{\rm eq}$(C) for others.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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