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4-(4-Bromophenyl)-3-methyl-1-phenyl-4,5-dihydro-1*H*-pyrazolo[3,4-*b*]pyridin-6(7*H*)-one

Feng Shi, Shu-Jiang Tu* and Song-Lei Zhu

Department of Chemistry, Xuzhou Normal University, Xuzhou 221116, People's Republic of China

Correspondence e-mail: laotu2001@263.net

The title compound, $C_{19}H_{16}BrN_3O$, was synthesized by the reaction of 5-amino-3-methyl-1-phenylpyrazole with 4-bromophenylaldehyde and Medrum's acid in glycol under microwave irradiation. X-ray crystal structure analysis reveals three crystallographically independent molecules in the asymmetric unit. In all these molecules, the dihydropyridine ring adopts a distorted envelope conformation. In the crystal structure, the molecules exist as N—H···O hydrogen-bonded dimers.

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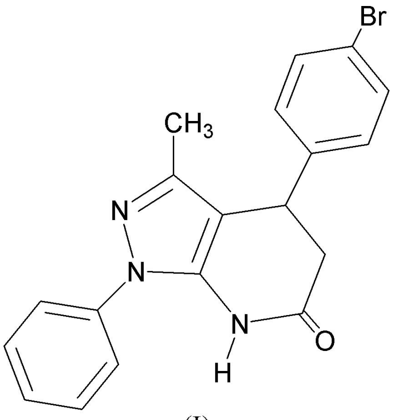
Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.009 Å
R factor = 0.079
wR factor = 0.157
Data-to-parameter ratio = 13.8

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 asymmetric unit of (I) contains three crystallographically independent molecules (Fig. 1). The corresponding bond distances and angles agree with one another (Table 1). In all the three molecules, the dihydropyridine ring adopts a distorted envelope conformation. The dihedral angles between the planes of the pyrazole and the attached phenyl rings are 41.5 (2), 40.1 (2) and 46.4 (2)°. The crystal packing shows that the molecules exist as N—H···O hydrogen-bonded (Table 2) dimers. The molecular packing is further stabilized by C—H···Br interactions (Fig. 2).

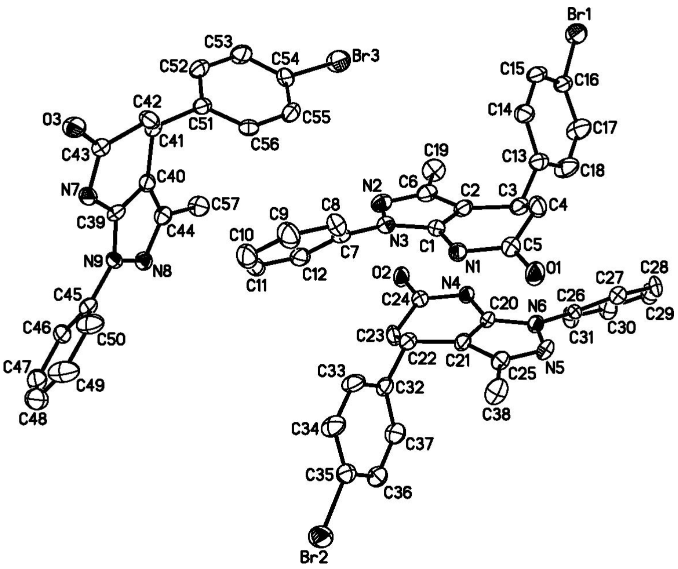


Figure 1
The asymmetric unit of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted.

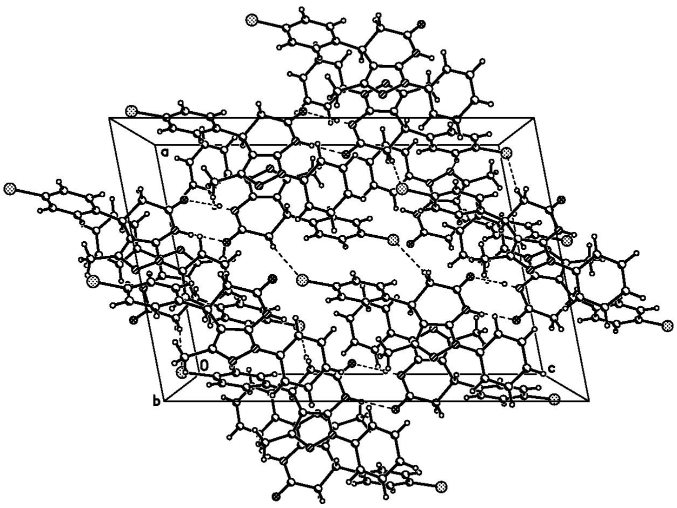


Figure 2
The molecular packing of (I), viewed along the *b* axis. Dashed lines indicate hydrogen bonds.

Experimental

Compound (I) was prepared by the reaction of 5-amino-3-methyl-1-phenylpyrazole (2 mmol) with 4-bromophenylaldehyde (2 mmol) and Medrum's acid (2 mmol) in glycol (1 ml) under microwave irradiation for 5 min (yield 82%, m.p. 505–506 K). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution (95%).

Crystal data

$C_{19}H_{16}BrN_3O$
$M_r = 382.26$
Monoclinic, $P2_1/c$
$a = 13.905\ (2)$ Å
$b = 17.965\ (3)$ Å
$c = 20.505\ (4)$ Å
$\beta = 101.026\ (7)^\circ$
$V = 5027.5\ (15)$ Å^3
$Z = 12$

$D_x = 1.515$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 19 389 reflections
$\theta = 3.0$–25.3°
$\mu = 2.46$ mm^{-1}
$T = 193\ (2)$ K
Block, colourless
$0.60 \times 0.40 \times 0.39$ mm

Data collection

Rigaku Mercury CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min} = 0.274$, $T_{\max} = 0.383$
48 280 measured reflections

9180 independent reflections
7942 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$
$\theta_{\max} = 25.4^\circ$
$h = -16 \rightarrow 16$
$k = -20 \rightarrow 21$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.079$
$wR(F^2) = 0.157$
$S = 1.13$
9180 reflections
665 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0145P)^2 + 35.9447P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.91$ e Å^{-3}
$\Delta\rho_{\min} = -0.98$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.238 (7)	N5—N6	1.388 (7)
O2—C24	1.229 (7)	N6—C20	1.352 (7)
O3—C43	1.224 (7)	N6—C26	1.417 (7)
N1—C5	1.365 (7)	N7—C43	1.368 (7)
N1—C1	1.396 (7)	N7—C39	1.398 (7)
N2—C6	1.329 (8)	N8—C44	1.330 (7)
N2—N3	1.375 (7)	N8—N9	1.373 (6)
N3—C1	1.365 (7)	N9—C39	1.357 (7)
N3—C7	1.403 (7)	N9—C45	1.433 (7)
N4—C24	1.355 (7)	C1—C2	1.343 (8)
N4—C20	1.401 (7)	C20—C21	1.365 (8)
N5—C25	1.324 (7)	C39—C40	1.357 (8)
C2—C3—C13—C18	−117.5 (7)	C21—C22—C32—C37	−58.7 (8)
C2—C3—C13—C14	56.1 (8)	C40—C41—C51—C52	119.4 (6)
C21—C22—C32—C33	117.3 (6)	C40—C41—C51—C56	−54.9 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1$^{\rm i}$	0.88 (1)	1.94 (1)	2.812 (6)	172 (5)
N4—H4···O3$^{\rm ii}$	0.88 (1)	1.94 (2)	2.814 (6)	173 (8)
N7—H7···O2$^{\rm iii}$	0.88 (1)	1.96 (1)	2.834 (6)	174 (6)
C4—H4*A*···Br3$^{\rm iv}$	0.99	2.84	3.826 (7)	173

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

The H atoms of the NH groups were located in a difference Fourier map and were refined isotropically, with an N—H distance restraint of 0.88 (1) Å. 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 $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and 1.2 $U_{\rm eq}({\rm C})$ for others. The highest residual peak is located 0.92 Å from atom Br2.

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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