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6-Amino-4-[4-(dimethylamino)phenyl]-3-methyl-1-phenylpyrazolo[3,4-*b*]pyridine-5-carbonitrile

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.103
Data-to-parameter ratio = 12.4

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

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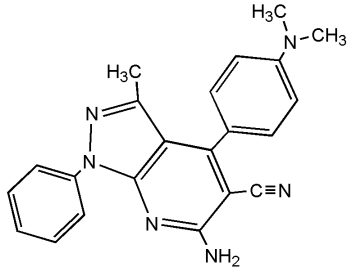
The title compound, $C_{22}H_{20}N_6$, was synthesized by the reaction of 5-amino-3-methyl-1-phenylpyrazole with [4-(dimethylamino)benzylidene]malononitrile in glycol under microwave irradiation. X-ray crystal structure analysis reveals that the molecules exist as centrosymmetric N—H···N hydrogen-bonded dimers.

Comment

The pyrazolo[3,4-*b*]pyridine system has many interesting biological and pharmacological properties, such as active antitubercular action and action against Gram-positive and -negative bacteria, and is used in the treatment of a wide variety of stress-related illnesses (Sekikawa *et al.*, 1973; Kuczynski *et al.*, 1979; El-Dean *et al.*, 1991; Chen, 1995). As part of our programme aimed at employing microwave irradiation for the preparation of heterocyclic compounds (Tu *et al.*, 2004), we have recently synthesized pyrazolo[3,4-*b*]-pyridine derivatives under microwave irradiation. Here, we report the crystal structure of the title compound, (I).



(I)

The dihedral angle between the pyridine and pyrazole planes is 2.9 (1)°, indicating that they are almost coplanar (Fig. 1). The C7–C12 phenyl ring forms a dihedral angle of 66.48 (5)° with the pyrazole ring, and the dihedral angle between the pyridine and dimethylaminophenyl rings is 49.30 (5)°.

In the crystal structure, the molecules of (I) form centrosymmetric N—H···N hydrogen-bonded dimers (Table 1 and Fig. 2).

Experimental

Compound (I) was prepared by the reaction of 5-amino-3-methyl-1-phenylpyrazole (2 mmol) with [4-(dimethylamino)benzylidene]-malononitrile (2 mmol) in glycol (1 ml) under microwave irradiation (yield 91%; m.p. 507 K). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution (95%).

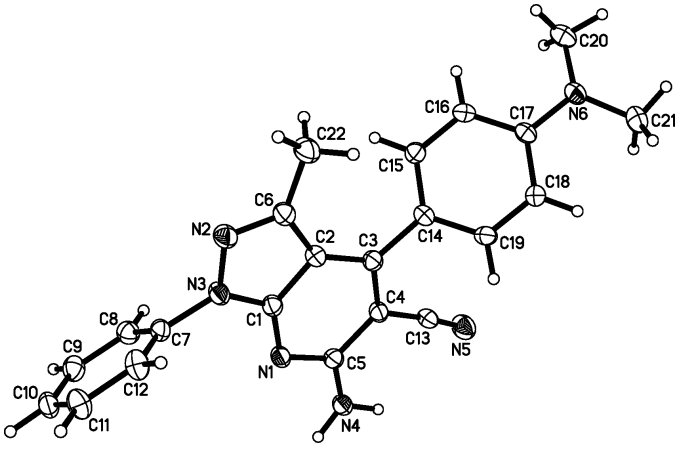


Figure 1
The molecular structure of (I), showing 45% probability displacement ellipsoids and the atom-numbering scheme.

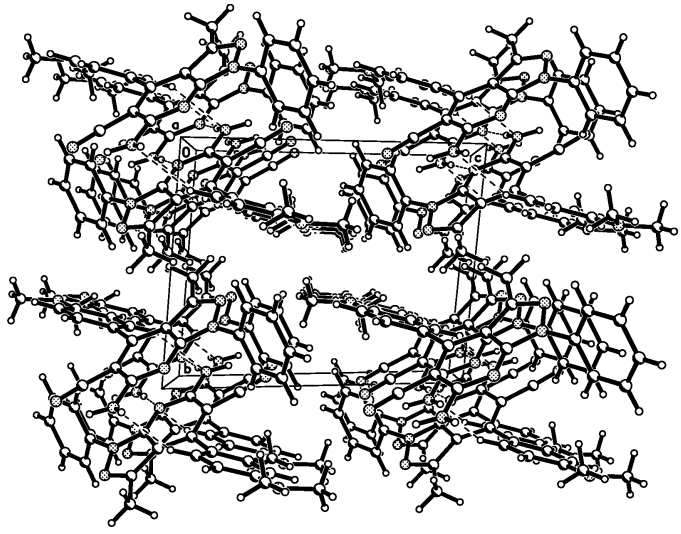


Figure 2
The molecular packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

Crystal data

$C_{22}H_{20}N_6$
M_r = 368.44
Triclinic, $P\overline{1}$
a = 8.493 (3) Å
b = 9.446 (3) Å
c = 12.144 (4) Å
α = 91.367 (5)°
β = 109.463 (7)°
γ = 100.148 (5)°
V = 900.7 (5) Å^3
Z = 2
D_x = 1.359 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3551 reflections
θ = 3.1–25.3°
μ = 0.09 mm^{-1}
T = 193 (2) K
Block, light yellow
0.69 × 0.25 × 0.12 mm

Data collection

Rigaku Mercury CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.944, T_{max} = 0.990
8996 measured reflections
3288 independent reflections
2856 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.4°
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 1.07
3288 reflections
265 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2 + 0.2172P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4B···N1^{i}	0.90 (2)	2.21 (2)	3.110 (2)	177 (2)

Symmetry code: (i) $-1-x, -y, -z$.

The H atoms of the amino group were located in a difference Fourier map and were refined isotropically [N—H = 0.90 (2) and 0.91 (2) 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 0.95–0.98 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for others.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2000–2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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