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2-Amino-4-(4-methoxyphenyl)-6-(piperidin-1-yl)-pyridine-3,5-dicarbonitrile

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Key indicators

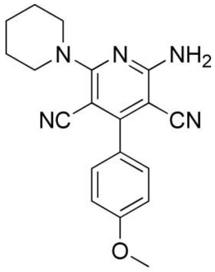
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.095
Data-to-parameter ratio = 8.8

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

The crystal structure of the title compound, $C_{19}H_{19}N_5O$, is stabilized by intermolecular C—H···O hydrogen bonds and C—H···π interactions.

Comment

The structure of the title compound, (I) (Fig. 1), is similar to that of 2-amino-4-(4-methoxyphenyl)-6-(pyrrolidin-1-yl)pyridine-3,5-dicarbonitrile (Raghukumar *et al.*, 2003). The methoxyphenyl ring is twisted with respect to the pyridine ring, with a dihedral angle of 43.20 (1)° between them. The piperidine ring adopts a half-chair conformation. The crystal packing is consolidated by intermolecular C—H···O hydrogen bonds and C—H···π interactions (Table 1 and Fig. 2).



(I)

Experimental

Piperidine (0.38 g, 4.4 mmol) was added dropwise to a solution of a 4-methoxybenzaldehyde (0.50 g, 3.67 mmol) and malononitrile (0.50 g,

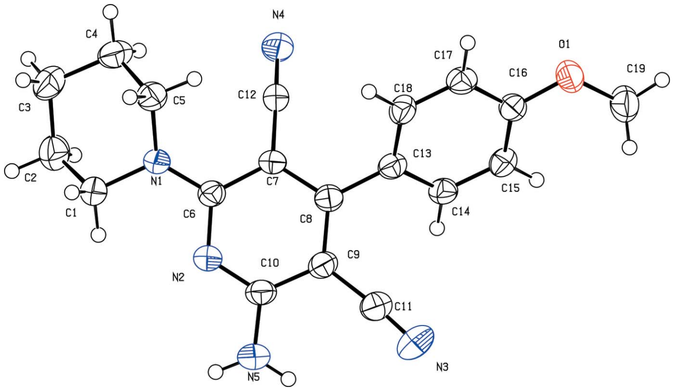


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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7.35 mmol) in 5 ml of anhydrous methanol at 273 K. The reaction mixture was then stirred at room temperature for 10 h. The resulting mixture was purified by chromatography on silica using petroleum ether/acetone (5:1*v*/*v*) as eluant to obtain the title compound as a gray–yellow solid (yield 0.39 g, 33%). The product was recrystallized from ethanol at room temperature to give crystals suitable for single-crystal X-ray diffraction.

Crystal data

$C_{19}H_{19}N_5O$
$M_r = 333.39$
Monoclinic, $P2_1$
$a = 9.4430$ (7) Å
$b = 7.5675$ (6) Å
$c = 12.5714$ (9) Å
$\beta = 112.06^\circ$
$V = 832.58$ (11) Å^3
$Z = 2$
$D_x = 1.330$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.18 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{\min} = 0.985$, $T_{\max} = 0.991$
9714 measured reflections
2051 independent reflections
1618 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.144$
$\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.095$
$S = 0.94$
2051 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···O1$^{\rm i}$	0.97	2.51	3.228 (3)	131
C19—H19*C*···*Cg*3$^{\rm ii}$	0.96	2.72	3.345 (4)	123

Symmetry codes: (i) $x - 1, y, z - 1$; (ii) $-x + 1, y - \frac{1}{2}, -z + 1$. *Cg*3 .is the centroid of the benzene ring

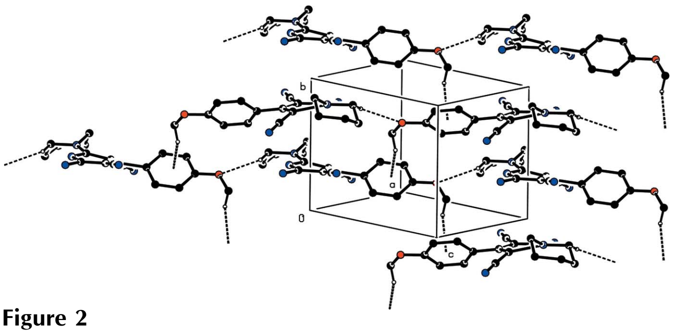

Figure 2
Part of the packing of (I), with hydrogen bonds and C—H···π interactions drawn as dashed lines. For clarity, H atoms not involved in these interactions have been omitted.

We tried to obtain crystals suitable for single-crystal X-ray diffraction from three different solvents. Unfortunately, the single crystals were all of poor quality, hence the high value of $R_{\rm int}$. In the absence of significant anomalous dispersion effects, 1733 Friedel pairs were merged. H atoms bound to N5 were located in a difference Fourier map and refined with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$ and the restraint N—H = 0.86 (1) Å. Other H atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å, $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$ (C) for aromatic, C—H = 0.97 Å, $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for CH_2, and C—H = 0.96 Å, $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for CH_3 atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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