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2,6-Diamino-4-(4-methoxyphenyl)-1,4-dihydropyridine-3,5-dicarbonitrile *N*,*N*-dimethylformamide solvate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.127
Data-to-parameter ratio = 12.6

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

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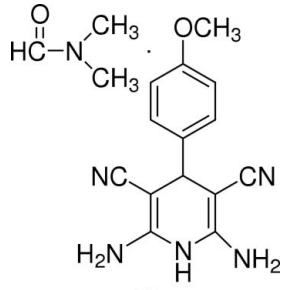
The title compound, $C_{14}H_{11}N_5O\cdot C_3H_7NO$, was synthesized by the reaction of 4-methoxybenzaldehyde with malononitrile and ammonium acetate under microwave irradiation. The dihedral angle between the pyridine and benzene planes is 56.07 (8)°.

Comment

3,5-Dicyanopyridine derivatives exhibit a wide range of bioactivities, such as antifungal, insecticidal, herbicidal, miticidal, nematocidal and antimicrobial activity (Gante & Lust, 1971). Some substituted 3,5-dicyanopyridines have recently been reported to exhibit a high conductance-type calcium-activated K-channel opening effect (Hirochika *et al.*, 2003) and to be adenosine receptor-selective ligands (Rosentreter *et al.*, 2002), which are useful in the treatment of many diseases. More importantly, they are also versatile intermediates in organic synthesis (Castedo *et al.*, 1984). As a consequence, much attention has been paid to the synthesis of these derivatives during the past 50 years. We report here the crystal structure of the title compound, (I).



(I)

The 2,6-diamino-1,4-dihydropyridine-3,5-dicarbonitrile fragment of (I) (atoms C8–C14/N1–N5) is almost planar, with an r.m.s. deviation of 0.076 Å (Fig. 1). The dihedral angle between the pyridine and benzene planes is 56.07 (8)°.

The crystal packing shows that intermolecular N—H···O, N—H···N, C—H···O and C—H···N hydrogen bonds (Table 1) form a three-dimensional network (Fig. 2).

Experimental

Compound (I) was prepared by the reaction of 4-methoxybenzaldehyde (1 mmol) with malononitrile (2 mmol) and ammonium acetate (1 mmol) under microwave irradiation for 4 min (yield 95%; m.p. 573 K). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol and *N*,*N*-dimethylformamide solution (5:1 *v*/*v*).

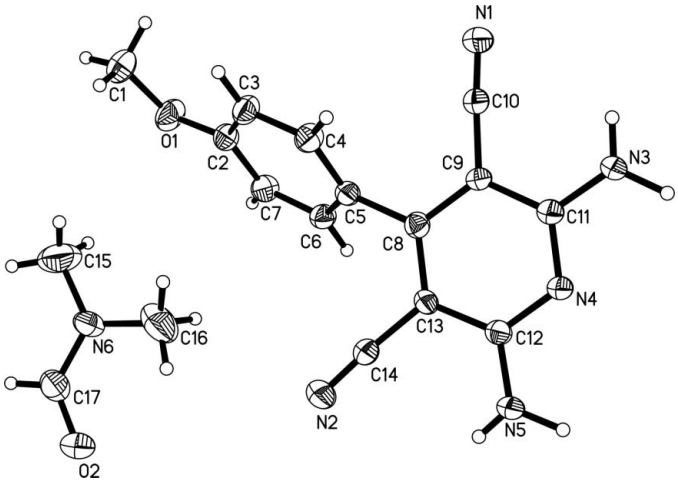


Figure 1
The structure (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

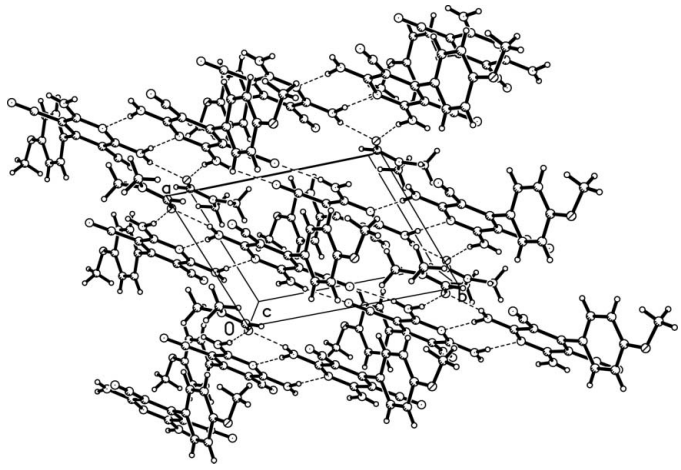


Figure 2
The molecular packing of (I), viewed approximately along the *c* axis. Dashed lines indicate hydrogen bonds.

Crystal data

$C_{14}H_{11}N_5O\cdot C_3H_7NO$	$Z = 2$
$M_r = 338.37$	$D_x = 1.272$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 6.9580$ (13) Å	Cell parameters from 1237 reflections
$b = 9.5297$ (18) Å	$\theta = 2.3$–$25.6°$
$c = 14.695$ (3) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 99.900$ (3)°	$T = 294$ (2) K
$\beta = 102.505$ (3)°	Plate, colourless
$\gamma = 106.527$ (3)°	0.22 × 0.18 × 0.06 mm
$V = 883.1$ (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3087 independent reflections
	1774 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.026$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -6 \rightarrow 8$
$T_{min} = 0.976$, $T_{max} = 0.995$	$k = -11 \rightarrow 11$
4506 measured reflections	$l = -14 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.1375P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.046$	
$wR(F^2) = 0.127$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.00$	$\Delta\rho_{max} = 0.18$ e Å^{-3}
3087 reflections	$\Delta\rho_{min} = -0.20$ e Å^{-3}
245 parameters	
H atoms treated by a mixture of independent and constrained refinement	

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O2^{i}	0.88 (3)	2.07 (3)	2.846 (3)	147 (2)
N3—H3B···N4^{ii}	1.00 (3)	2.04 (3)	3.033 (3)	168 (2)
N5—H5A···N2^{iii}	0.79 (3)	2.45 (3)	3.195 (3)	158 (2)
N5—H5B···O2^{iii}	0.98 (3)	2.13 (3)	3.083 (3)	166 (2)
C6—H6A···N1^{iv}	0.93	2.59	3.506 (4)	171
C7—H7A···O1^{v}	0.93	2.58	3.503 (4)	170
C17—H17A···N1^{vi}	0.93	2.48	3.288 (4)	145

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

The H atoms of the amino groups were located in a difference Fourier map and refined isotropically [N—H = 0.79 (3)–1.00 (3) Å]. All other H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H distances of 0.93 or 0.96 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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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