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2-Amino-4-(4-chlorophenyl)-6-morpholinopyridine-3,5-dicarbonitrile

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.095
Data-to-parameter ratio = 13.0

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

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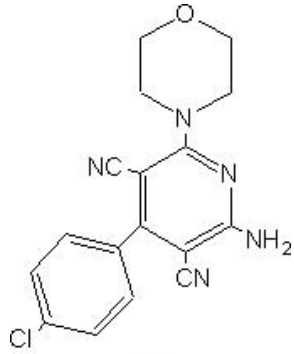
The title compound, $C_{17}H_{14}ClN_5O$, was synthesized by the reaction of malononitrile with 4-chlorobenzaldehyde and morpholine in glycol under microwave irradiation. X-ray analysis reveals that the morpholine ring is in a chair conformation. In the crystal structure, intermolecular N—H···O, C—H···N and N—H···N hydrogen bonds form a three-dimensional network.

Comment

3,5-Dicyanopyridine derivatives exhibit a wide range of bioactivities, such as antifungal, insecticidal, herbicidal, miticidal, nematocidal and antimicrobial activity (Gante & Lust, 1971). 2-Amino-6-substituted-amino-4-substituted-3,5-dicyanopyridines have recently been reported to exhibit a high conductance-type calcium-activated K channel opening effect (Hironori *et al.*, 2002) and to be adenosine receptor-selective ligands (Ulrich *et al.*, 2002), which are useful in the treatment of many diseases. 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 morpholine ring in (I) is in a chair conformation (Fig. 1). The dihedral angle between the pyridine and benzene planes is 66.37 (6)°. The crystal packing shows that intermolecular N—H···O, C—H···N and N—H···N hydrogen bonds (Table 1) form a three-dimensional network (Fig. 2). In addition, short intermolecular contacts exist between atoms Cl1 and N3($-x$, $1-y$, $1-z$) [3.151 (2) Å] and between Cl1 and O1($1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) [3.248 (2) Å].

Experimental

Compound (I) was prepared by the reaction of malononitrile (2 mmol) with 4-chlorobenzaldehyde (1 mmol) and morpholine

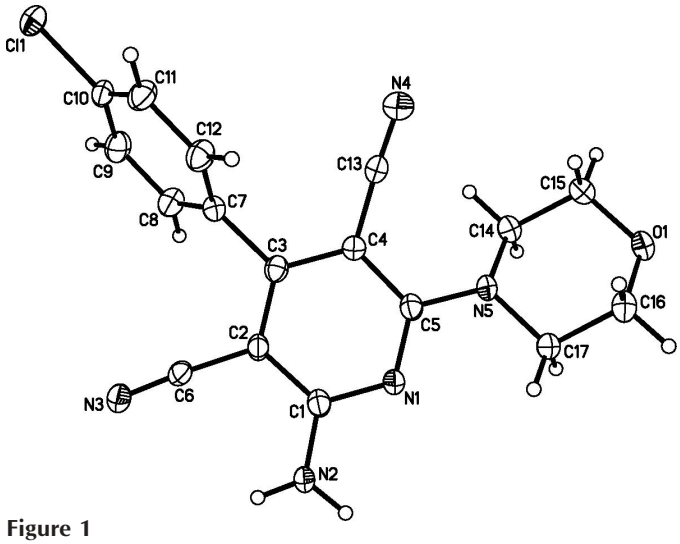


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

(1 mmol) in glycol (1 ml) under microwave irradiation for 4 min (yield 94%, m.p. 500–501 K). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{17}H_{14}ClN_5O$	D_x = 1.413 Mg m^{-3}
M_r = 339.78	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 5837 reflections
a = 7.0484 (15) Å	θ = 3.2–25.3°
b = 10.3371 (19) Å	μ = 0.25 mm^{-1}
c = 22.174 (4) Å	T = 193 (2) K
β = 98.547 (6)°	Block, light yellow
V = 1597.6 (5) Å^3	0.35 × 0.20 × 0.10 mm
Z = 4	

Data collection

Rigaku Mercury CCD area-detector diffractometer	2922 independent reflections
ω scans	2508 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (Jacobson, 1998)	R_{int} = 0.036
T_{min} = 0.917, T_{max} = 0.975	θ_{max} = 25.3°
15505 measured reflections	$h = -8 \rightarrow 8$
	$k = -12 \rightarrow 12$
	$l = -26 \rightarrow 26$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 0.7626P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.041	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.095	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
2922 reflections	
225 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
N2—H2A···N3^{i}	0.86 (2)	2.29 (2)	3.129 (2)	163 (2)
N2—H2B···O1^{ii}	0.91 (2)	2.04 (2)	2.940 (2)	170 (2)
C16—H16B···N4^{ii}	0.99	2.61	3.478 (3)	147

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

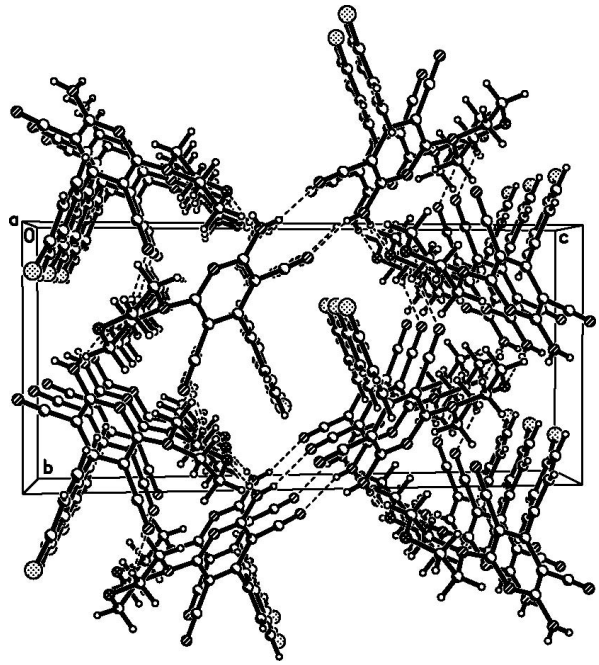


Figure 2
The molecular packing of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

The H atoms of the amine group were located in a difference Fourier map and were refined isotropically [N—H = 0.86 (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 0.95–0.99 Å and with U_{iso}(H) = 1.2U_{eq}(C).

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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References

Gante, J. & Lust, S. (1971). US Patent No. 3629270.
Hironori, H., Susumu, W., Tomofumi, T., Kenichi, K., Toshio, O., Yuusuke, H. & Chikashi, S. (2002). Japanese Patent No. 6237; *Chem. Abstr.* (2002), **136**, 118474.
Jacobson, R. (1998). Private communication to the Rigaku Corporation, Tokyo, Japan.
Rigaku (1999). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2003). *CrystalStructure*. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Ulrich, R., Thomas, K., Andrea, V., Walter, H., Nicole, D., Thomas, K., Klaus, D. & Stasch, J. P. (2002). Germany Patent No. 70520; *Chem. Abstr.* (2002), 137, 216881.