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2-Amino-6-(dimethylamino)pyridine-3,5-dicarbonitrile

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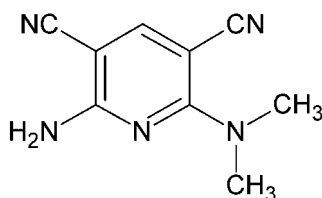
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.147; data-to-parameter ratio = 12.0.

The title compound, $\text{C}_9\text{H}_9\text{N}_5$, is slightly twisted from planarity, with a maximum deviation of 0.0285 (13) Å from the pyridine plane for the C atom bearing the amino group. The cyano groups are on different sides of the pyridine plane, with C- and N-atom deviations of 0.072 (3)/0.124 (4) and -0.228 (4)/ -0.409 (5) Å from the pyridine plane. In the crystal, $\text{N}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds connect the molecules into zigzag chains running along the c axis.

Related literature

For the synthesis of similar structures, see: Horton *et al.* (2012*a,b*); Soliman *et al.* (2012). For the biological significance of cyanoamino pyridines, see: Al-Haiza *et al.* (2003); Bhalerao & Krishnaiah (1995); Deo *et al.* (1990); Murata *et al.* (2003); Konda *et al.* (2010); Altomare *et al.* (2000); Hosni & Abdulla (2008); Shishoo *et al.* (1983).



Experimental

Crystal data

$\text{C}_9\text{H}_9\text{N}_5$
 $M_r = 187.21$
 Monoclinic, $C2/c$
 $a = 28.667$ (7) Å
 $b = 3.9702$ (10) Å
 $c = 17.950$ (4) Å
 $\beta = 112.920$ (3)°

$V = 1881.7$ (8) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 296$ K
 $0.32 \times 0.21 \times 0.03$ mm

Data collection

Bruker SMART 1000 CCD
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.972$, $T_{\max} = 0.997$

4846 measured reflections
 1658 independent reflections
 1173 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.147$
 $S = 1.03$
 1658 reflections
 138 parameters

H atoms treated by a mixture of
 independent and constrained
 refinement
 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H1}\cdots\text{N3}^{\text{i}}$	0.88 (3)	2.25 (3)	3.119 (3)	167 (2)
$\text{N2}-\text{H2}\cdots\text{N1}^{\text{ii}}$	0.88 (3)	2.43 (3)	3.260 (3)	158 (3)
$\text{C3}-\text{H3}\cdots\text{N4}^{\text{iii}}$	0.93	2.55	3.471 (4)	170

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97.

The authors are thankful to the University of Hong Kong for providing the single-crystal X-ray crystallography facility.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2369).

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supporting information

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2-Amino-6-(dimethylamino)pyridine-3,5-dicarbonitrile

Shaaban K. Mohamed, Ahmed M. Soliman, Eman M. M. Abdel-Raheem, Sohail Saeed and Wing-Tak Wong

S1. Comment

In continuation of our research interest in the synthesis of potential biologically active molecules (Soliman *et al.*, 2012; Horton *et al.*, 2012*a,b*), we got prompted to study the chemical and pharmacological characterization of new cyano-amino pyridine derivatives due to their vibrant chemical activities. Hence cyano-amino pyridines have been considered as convenient synthons due to their diverse applications particularly in organic synthesis (Shishoo *et al.*, 1983; Deo *et al.*, 1990; Bhalerao & Krishnaiah, 1995; Al-Haiza *et al.*, 2003) and medicinal chemistry (Altomare *et al.*, 2000; Hosni & Abdulla, 2008; Murata *et al.*, 2003; Konda *et al.*, 2010).

The title compound, 2-amino-6-(dimethylamino)-pyridine-3,5-dicarbonitrile, is slightly twisted. The maximum deviation from the mean plane of the pyridyl ring, N1/C1—C5 (marked with asterisk) is 0.0285 (13) Å. The cyano groups are flipped to different sides of the pyridine plane with atoms C6 & N3 showing deviations of +0.072 (3) Å and +0.124 (4) Å, while atoms C7 & N4 are bent out of the pyridine plane by -0.228 (4) Å and -0.409 (5) Å, respectively.

Hydrogen bonding interactions are observed in the crystal lattice connecting the molecules into zigzag chains running along the *c*-axis. As it is expected, N—H \cdots N interactions are shorter as the observed N4 \cdots H3(-C3) distance.

S2. Experimental

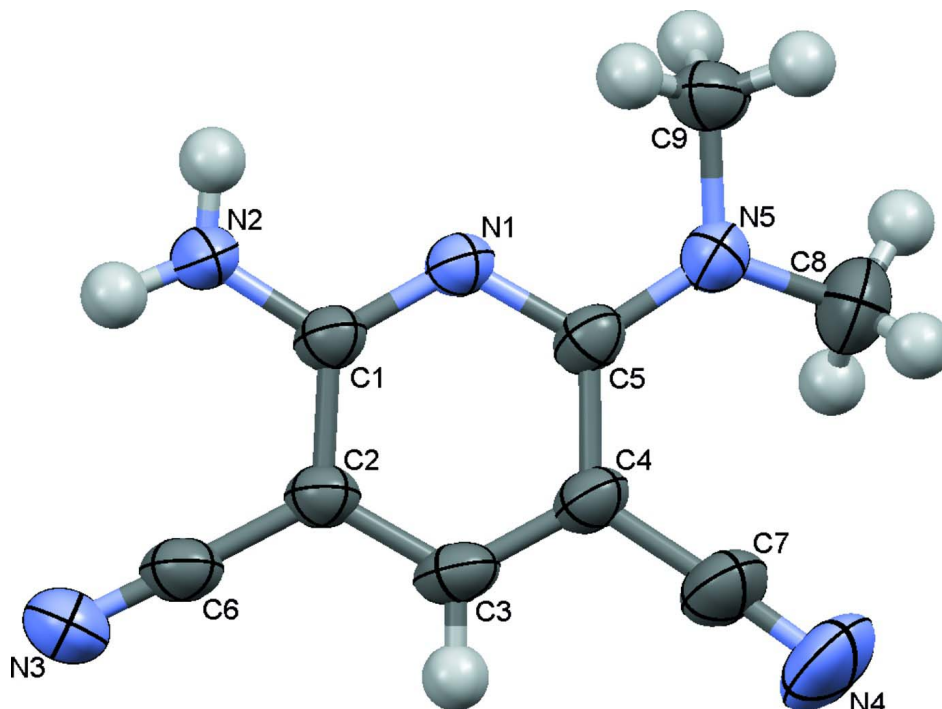
The title compound (1) was obtained as a by-product from the reaction of 2-amino-6-chloropyridine-3,5-dicarbonitrile (1 mmol; 179 mg) with amino guanidine (1 mmol; 74 mg) in dimethylformamide. The reaction mixture was refluxed for 4 h at 426 K and then poured on cold water. A solid product was filtered off, dried and recrystallized from ethanol to afford cupric needles which were suitable for X-Ray diffraction without further recrystallization. Yield 45% and m.p. 453 K.

S3. Refinement

The structure was solved by direct methods (*SHELXS97*, Sheldrick, 2008) and expanded using Fourier techniques. All non-H atoms were refined anisotropically.

C-bound H atoms are all placed at geometrical positions with C—H = 0.93 and 0.96 Å for phenyl and methyl H-atoms, respectively. C-bound phenyl hydrogen atoms are refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, methyl H-atoms are refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. N-bound H atoms were located from the difference Fourier map and were refined isotropically.

Highest peak is 0.16 at (0.1967, 0.2214, 0.0041) [1.04 Å from H8C] Deepest hole is -0.19 at (0.1226, 0.1471, 0.2613) [1.01 Å from C5]

**Figure 1**

Molecular structure of the title compound showing thermal ellipsoids on the 50% probability level.

2-Amino-6-(dimethylamino)pyridine-3,5-dicarbonitrile

Crystal data

$C_9H_9N_5$

$M_r = 187.21$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 28.667 (7) \text{ \AA}$

$b = 3.9702 (10) \text{ \AA}$

$c = 17.950 (4) \text{ \AA}$

$\beta = 112.920 (3)^\circ$

$V = 1881.7 (8) \text{ \AA}^3$

$Z = 8$

$F(000) = 784$

$D_x = 1.322 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4846 reflections

$\theta = 3.1\text{--}25.0^\circ$

$\mu = 0.09 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Plate, yellow

$0.32 \times 0.21 \times 0.03 \text{ mm}$

Data collection

Bruker SMART 1000 CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω and φ scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 2004)

$T_{\min} = 0.972$, $T_{\max} = 0.997$

4846 measured reflections

1658 independent reflections

1173 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.1^\circ$

$h = -33 \rightarrow 34$

$k = -4 \rightarrow 4$

$l = -21 \rightarrow 18$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.147$
 $S = 1.03$
 1658 reflections
 138 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0915P)^2 + 0.1839P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.07712 (6)	0.0877 (4)	0.27256 (9)	0.0417 (5)
N2	0.00997 (6)	0.2230 (6)	0.15638 (13)	0.0573 (6)
H1	-0.0050 (9)	0.284 (6)	0.1053 (17)	0.068 (8)*
H2	-0.0096 (12)	0.128 (8)	0.1777 (19)	0.094 (9)*
N3	0.05670 (8)	0.6406 (6)	0.02533 (12)	0.0669 (6)
N4	0.25896 (8)	0.0677 (9)	0.35083 (15)	0.1042 (10)
N5	0.14100 (6)	-0.0606 (5)	0.39210 (10)	0.0488 (5)
C1	0.06018 (7)	0.2187 (5)	0.19854 (12)	0.0415 (5)
C2	0.09339 (7)	0.3580 (5)	0.16489 (12)	0.0431 (5)
C3	0.14482 (7)	0.3269 (6)	0.20982 (13)	0.0499 (6)
H3	0.1676	0.4079	0.1887	0.060*
C4	0.16300 (7)	0.1796 (5)	0.28473 (13)	0.0463 (5)
C5	0.12699 (7)	0.0699 (5)	0.31732 (12)	0.0413 (5)
C6	0.07369 (8)	0.5132 (6)	0.08746 (14)	0.0498 (6)
C7	0.21622 (9)	0.1175 (7)	0.32283 (15)	0.0673 (7)
C8	0.18827 (9)	0.0208 (8)	0.45832 (15)	0.0782 (8)
H8A	0.1828	0.0356	0.5077	0.117*
H8B	0.2006	0.2328	0.4478	0.117*
H8C	0.2127	-0.1520	0.4634	0.117*
C9	0.10351 (8)	-0.2200 (6)	0.41700 (13)	0.0561 (6)
H9A	0.1197	-0.3882	0.4572	0.084*
H9B	0.0778	-0.3238	0.3710	0.084*
H9C	0.0884	-0.0530	0.4392	0.084*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0341 (9)	0.0539 (10)	0.0397 (10)	-0.0009 (7)	0.0172 (7)	-0.0014 (8)
N2	0.0344 (10)	0.0948 (16)	0.0427 (12)	-0.0056 (9)	0.0150 (9)	0.0117 (11)
N3	0.0644 (13)	0.0872 (15)	0.0559 (13)	-0.0010 (11)	0.0308 (11)	0.0131 (12)
N4	0.0412 (12)	0.175 (3)	0.0971 (19)	0.0216 (15)	0.0270 (12)	-0.0016 (19)
N5	0.0376 (9)	0.0639 (11)	0.0435 (10)	0.0045 (8)	0.0142 (8)	0.0037 (9)
C1	0.0364 (10)	0.0501 (12)	0.0411 (12)	-0.0021 (8)	0.0186 (9)	-0.0048 (9)
C2	0.0404 (11)	0.0522 (12)	0.0425 (12)	-0.0026 (9)	0.0224 (9)	-0.0020 (10)
C3	0.0419 (12)	0.0610 (13)	0.0572 (14)	-0.0054 (9)	0.0307 (11)	-0.0060 (11)
C4	0.0332 (11)	0.0580 (13)	0.0515 (13)	0.0009 (9)	0.0205 (9)	-0.0041 (11)
C5	0.0348 (10)	0.0455 (11)	0.0449 (12)	0.0010 (8)	0.0169 (9)	-0.0067 (9)
C6	0.0468 (12)	0.0611 (14)	0.0509 (14)	-0.0040 (10)	0.0292 (11)	-0.0023 (12)
C7	0.0445 (14)	0.0956 (19)	0.0682 (17)	0.0067 (12)	0.0288 (12)	-0.0019 (14)
C8	0.0492 (14)	0.108 (2)	0.0642 (17)	0.0041 (14)	0.0074 (12)	0.0105 (16)
C9	0.0532 (13)	0.0662 (14)	0.0575 (14)	0.0078 (11)	0.0310 (12)	0.0106 (12)

Geometric parameters (\AA , $^\circ$)

N1—C1	1.330 (2)	C2—C6	1.421 (3)
N1—C5	1.341 (2)	C3—C4	1.370 (3)
N2—C1	1.340 (3)	C3—H3	0.9300
N2—H1	0.88 (3)	C4—C7	1.429 (3)
N2—H2	0.88 (3)	C4—C5	1.438 (3)
N3—C6	1.146 (3)	C8—H8A	0.9600
N4—C7	1.146 (3)	C8—H8B	0.9600
N5—C5	1.346 (3)	C8—H8C	0.9600
N5—C8	1.449 (3)	C9—H9A	0.9600
N5—C9	1.459 (3)	C9—H9B	0.9600
C1—C2	1.423 (3)	C9—H9C	0.9600
C2—C3	1.383 (3)		
C1—N1—C5	120.56 (16)	C7—C4—C5	123.7 (2)
C1—N2—H1	124.9 (15)	N1—C5—N5	116.88 (16)
C1—N2—H2	118 (2)	N1—C5—C4	120.45 (18)
H1—N2—H2	116 (3)	N5—C5—C4	122.66 (18)
C5—N5—C8	123.56 (19)	N3—C6—C2	178.3 (2)
C5—N5—C9	120.21 (17)	N4—C7—C4	177.7 (3)
C8—N5—C9	114.24 (18)	N5—C8—H8A	109.5
N1—C1—N2	117.60 (18)	N5—C8—H8B	109.5
N1—C1—C2	122.11 (18)	H8A—C8—H8B	109.5
N2—C1—C2	120.28 (19)	N5—C8—H8C	109.5
C3—C2—C6	122.45 (17)	H8A—C8—H8C	109.5
C3—C2—C1	117.08 (19)	H8B—C8—H8C	109.5
C6—C2—C1	120.45 (17)	N5—C9—H9A	109.5
C4—C3—C2	121.48 (18)	N5—C9—H9B	109.5
C4—C3—H3	119.3	H9A—C9—H9B	109.5

C2—C3—H3	119.3	N5—C9—H9C	109.5
C3—C4—C7	118.05 (19)	H9A—C9—H9C	109.5
C3—C4—C5	118.02 (18)	H9B—C9—H9C	109.5

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N2—H1...N3 ⁱ	0.88 (3)	2.25 (3)	3.119 (3)	167 (2)
N2—H2...N1 ⁱⁱ	0.88 (3)	2.43 (3)	3.260 (3)	158 (3)
C3—H3...N4 ⁱⁱⁱ	0.93	2.55	3.471 (4)	170

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x, y, -z+1/2$; (iii) $-x+1/2, y+1/2, -z+1/2$.