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Acta Cryst. (2017). E73, 1021–1025



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Crystal structures of *N'*-aminopyridine-2-carboximidamide and *N'*-{[1-(pyridin-2-yl)ethylidene]amino}pyridine-2-carboximidamide

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Received 13 April 2017

Accepted 6 June 2017

Edited by G. S. Nichol, University of Edinburgh, Scotland

Keywords: crystal structure; pyridine-2-carboximidamide; *N'*-{[1-(pyridin-2-yl)ethylidene]amino}pyridine-2-carboximidamide.

CCDC references: 1554569; 1554568

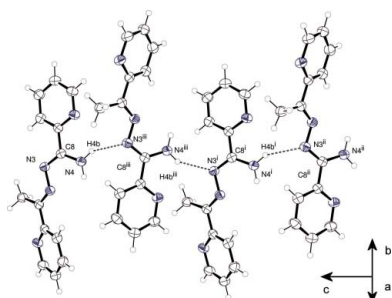
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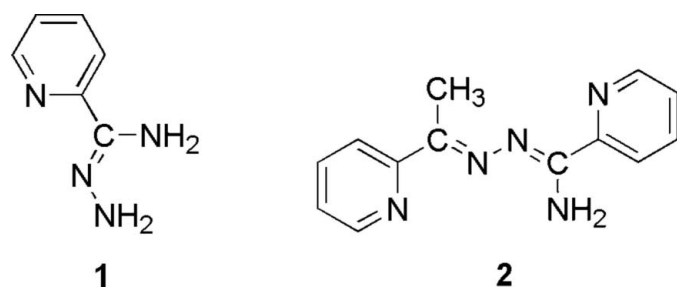
The crystal structures of *N'*-aminopyridine-2-carboximidamide (C₆H₈N₄), **1**, and *N'*-{[1-(pyridin-2-yl)ethylidene]amino}pyridine-2-carboximidamide (C₁₃H₁₃N₅), **2**, are described. The non-H atoms in compound **1** are nearly planar (r.m.s. deviation from planarity = 0.0108 Å), while **2** is twisted about the central N—N bond by 17.8 (2)°. Both molecules are linked by intermolecular N—H···N hydrogen-bonding interactions; **1** forms a two-dimensional hydrogen-bonding network and for **2** the network is a one-dimensional chain. The bond lengths of these molecules are similar to those in other literature reports of azine and diimine systems.

1. Chemical context

The preparation of hydrazidines with the general formula $RC(=NH)NHNH_2$ is accomplished by the action of hydrazine on the corresponding thioamide, imido ether or nitrile (Case, 1965). A pyridine-2-carboxamidrazide co-crystal form has previously been crystallized as a pyridine-2-carboxamidrazonium hydrogenoxalate salt, obtained by the reaction of pyridine-2-carboxamidrazide with oxalic acid (Wang *et al.*, 2007). Related molecules with diazine (N—N) bridges, obtained by condensation of hydrazidines with ketones can bring two metal centres into close proximity and provide an intramolecular exchange pathway for spin-exchange interactions *via* the *p*-orbital system (σ pathway) of the heterocyclic ligand (Xu *et al.*, 1997, 2000). The latter type of molecules present an unusual arrangement of potential donor sites, with many possible mononucleating and dinucleating coordination modes (Xu *et al.*, 1997). Semi-empirical structural calculations demonstrate that the N—N bond in these azines is rotationally soft, thereby allowing significant twisting at little energy cost (Kesslen *et al.*, 1999). Copper azine and imine complexes possess a significant antimalarial and anti-tumor action (Gokhale *et al.*, 2001*a,b*, 2003). Coordination complexes of 2-acetylpyridine-pyridine-2-carboxamidrazone have been obtained with cadmium(II), copper(II), nickel(II) and manganese(II) ions. The organic molecule behaves as a mono- and bis(bidentate) chelator (Xu *et al.*, 2000; Gokhale *et al.*, 2001*a*; Yue *et al.*, 2004, 2006). A polymorph of 2-acetylpyridine-pyridine-2-carboxamidrazone as been obtained with two crystallographically independent molecules included in the asymmetric unit (Yue *et al.*, 2006).



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2. Structural commentary

The molecular structure of **1** is shown in Fig. 1. The molecule is close to planar; the r.m.s. deviation of non-hydrogen atoms from planarity is 0.0108 Å with atom N2 displaying the largest deviation from the mean plane of 0.016 (3) Å. The geometry about N2 and N4 is not planar. H2A and H2B lie 0.12 (6) and 0.24 (6) Å out of the mean plane of non-hydrogen atoms. For H4A and H4B, the deviation is even greater at 0.37 (5) and 0.54 (5) Å from the mean plane. Rotation of the non-planar NH₂ group, particularly for N4, facilitates hydrogen bonding to other molecules. The N–N single bond length in **1** [1.424 (5) Å] is slightly shorter than that in the free hydrazine (1.449 Å).

The molecular structure of **2** is shown in Fig. 2. The molecule is not planar, perhaps as a result of conjunction of supramolecular interactions and packing effects. Each of the two ring systems is essentially planar (r.m.s. deviations for the two six-membered rings are 0.0162 and 0.0057 Å for N1/C1–C5 and N5/C9–C13, respectively). The hydrazidine group N3/C8/N4 is rotated slightly away from the plane of the six-membered ring along the C8–C9 bond by 8.6 (3)°. The imine group N2/C6/C7 is rotated from the plane of the adjacent six-membered ring by rotation about C5–C6 by 14.5 (2)°. The molecule is further distorted away from planarity by rotation of 17.8 (2)° about the central N2–N3 bond.

The bond lengths indicate that within the central chain of the molecule, the C6–N2 and C8–N3 linkages have largely

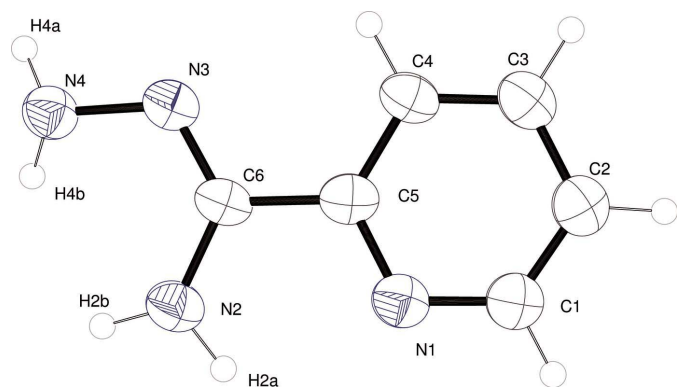


Figure 1
ORTEP representation of the asymmetric unit of **1**, with displacement ellipsoids drawn at the 50% probability level.

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N2–H2B···N4 ⁱ	0.95 (3)	2.16 (3)	3.106 (5)	174 (4)
N4–H4B···N4 ⁱ	0.94 (3)	2.51 (3)	3.357 (6)	149 (4)
N4–H4A···N3 ⁱⁱ	0.95 (3)	2.19 (4)	3.113 (5)	162 (4)

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

Table 2
Hydrogen-bond geometry (Å, °) for **2**.

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N4–H4B···N3 ⁱ	0.88	2.42	3.206 (3)	149

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

double-bond character. The azine linkages are in the *E,E* conformation, suggesting conjugation throughout the π systems. The C6–N2–N3 and C8–N3–N2 angles of 115.5 (2)° and 110.57 (19)°, respectively are significantly below the ideal sp^2 value of 120°, a consequence of the repulsion between the nitrogen lone pair and the adjacent bonds. The C6–N2–N3–C8 torsion angle is -162.2 (2)°. This large deviation from planarity has two consequences. First, there is a loss of conjugation between the imine bonds across the azine bond, reflected in the shorter imine bond length. The torsion also leads to a shorter N2–N3 bond length [1.408 (3) Å] compared to that observed for **1** [1.424 (5) Å]. Finally, a short intramolecular contact between N3ⁱ and H4B, 2.42 (3) Å, may add a favorable electrostatic contribution to the stability of this conformation. Notably, there is minimal change in the bond lengths within the ligands when a first row transition metal ion is bound. When the ligand chelates to a metal ion through both N3 and N5, only the bond length C8–N4 changes significantly, becoming shorter on binding.

3. Supramolecular features

There are two molecules of **1** in each unit cell and these are related by the screw axis. Curiously, N1 does not act as a hydrogen-bond acceptor. H2A is also not involved with the formation of any (short) classical hydrogen bonds. H2B forms a hydrogen bond to N4ⁱ [symmetry code: (i) $1 - x, y + \frac{1}{2}, 1 - z$].

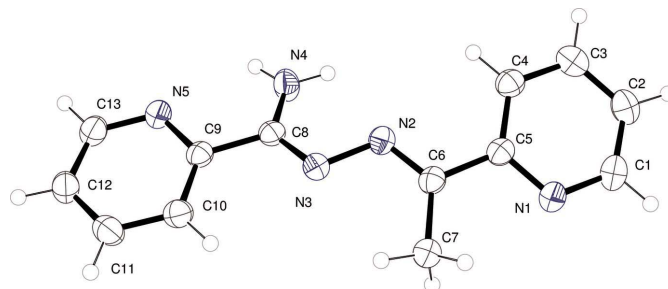


Figure 2
ORTEP representation of the asymmetric unit of **2**, with displacement ellipsoids drawn at the 50% probability level.

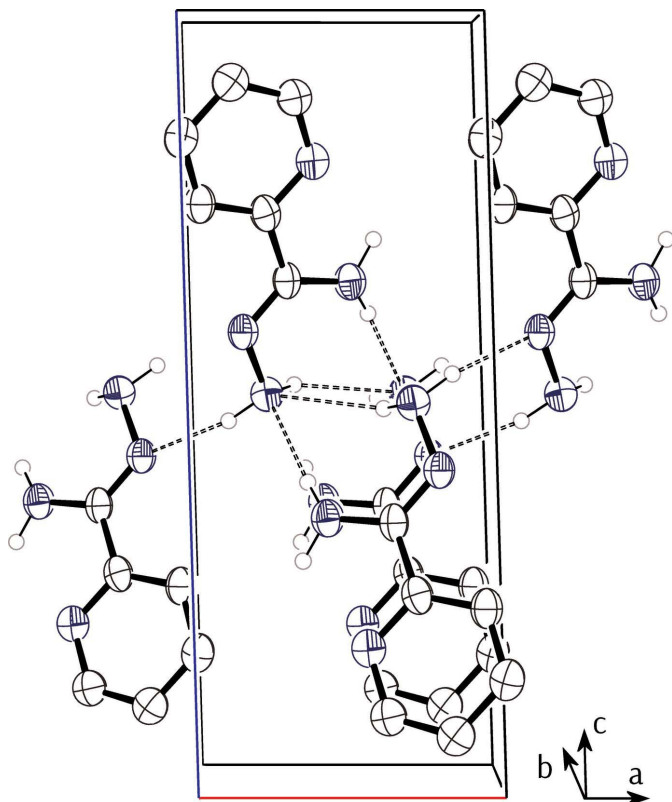


Figure 3
A portion of the hydrogen-bonded sheet present in **1**. Hydrogen bonds are shown as dashed lines.

This is augmented by the longer hydrogen bond $N4-H4B \cdots N4^i$. $N4-H4A$ forms a hydrogen bond to $N3^{ii}$ [symmetry code: (ii) $-x, y + \frac{1}{2}, -z + 1$]. These three sets of hydrogen bonds (Table 1) are sufficient to hold pairs of molecules together within the unit cell and to knit these dimers together to form sheets in the xy plane (see Fig. 3). These sheets then stack parallel to the $[001]$ direction, presumably held together by van der Waals interactions.

The classical hydrogen bonding (Table 2) in **2** is more sparse than in **1**. $N1$, $N2$, and $N5$ do not act as classical hydrogen-bond acceptors. A single symmetry-independent hydrogen bond [$N4-H4B \cdots N3^i$ [symmetry code: (i) $1/2 - x, 1 - y, z - 1/2$] is present and this knits the molecules of **2** together to form hydrogen-bonded chains along the $[001]$ direction, as shown in Fig. 4. There are subsidiary short $C-H \cdots N$ (pyridine) distances suggestive of intermolecular interactions.

4. Database survey

For literature on *N'*-aminopyridine-2-carboximidamide and related molecules, see Case *et al.* (1965). For the synthesis of *N'*-[[1-(pyridin-2-yl)ethylidene]amino]pyridine-2-carboximidamide and analogues, see Gokhale *et al.* (2001a,b, 2003) and Xu *et al.* (1997, 2000). For the coordination chemistry of *N'*-aminopyridine-2-carboximidamide, see Xu *et al.* (2000), Gokhale *et al.* (2001a) and Yue *et al.* (2004, 2006).

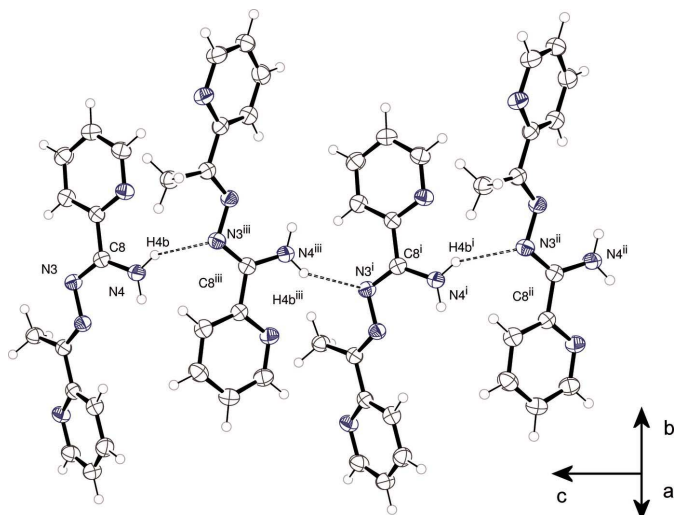


Figure 4
A portion of the hydrogen-bonded chain present in **2**. Hydrogen bonds are shown as dashed lines. Symmetry codes: (i) $x, y, z - 1$; (ii) $\frac{1}{2} - x, 1 - y, z - \frac{3}{2}$; (iii) $\frac{1}{2} - x, 1 - y, z - \frac{1}{2}$.

5. Synthesis and crystallization

The synthesis of *N'*-aminopyridine-2-carboximidamide and *N'*-[[1-(pyridin-2-yl)ethylidene]amino]pyridine-2-carboximidamide is depicted in Fig. 5.

N'-Aminopyridine-2-carboximidamide (**1**) was prepared by an analogy of the procedure published by Case (1965) with some modifications. A mixture of 2-cyanopyridine (0.05 mol), absolute ethanol (9 ml), and 95% hydrazine (15 ml) was stirred at room temperature for 2 h. The solid product was then dried under vacuum and recrystallized from benzene. *N'*-[[1-(Pyridin-2-yl)ethylidene]amino]pyridine-2-carboximidamide (**2**) was synthesized by an analogy of the procedure published by Gokhale *et al.* (2001a) by refluxing pyridine-2-carboxamidrazide (**1**) (0.5 g, 3.6 mmol) with excess 2-acetyl pyridine (0.5 g, 4.1 mmol) in absolute ethanol (20 ml) for 2 h. On cooling the product separates out in one week as yellow crystals which were filtered and dried.

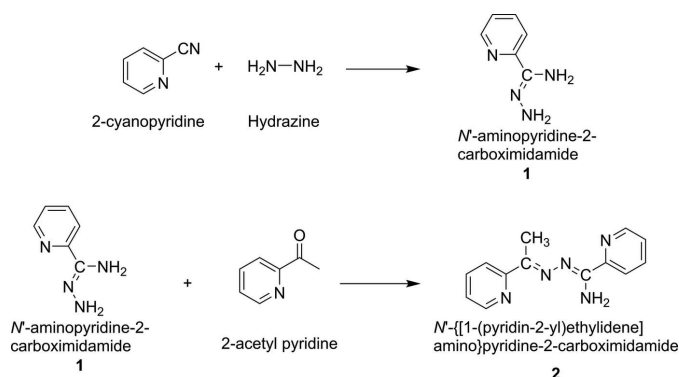


Figure 5
The synthesis of **1** and **2**.

Table 3
Experimental details.

	1	2
Crystal data		
Chemical formula	C ₆ H ₈ N ₄	C ₁₃ H ₁₃ N ₅
<i>M_r</i>	136.16	239.28
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁	Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁
Temperature (K)	150	150
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.6955 (14), 3.8408 (5), 14.592 (4)	6.6899 (5), 18.930 (2), 9.6561 (11)
α , β , γ (°)	90, 91.631 (19), 90	90, 90, 90
<i>V</i> (Å ³)	319.08 (12)	1222.8 (2)
<i>Z</i>	2	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.10	0.08
Crystal size (mm)	0.48 × 0.21 × 0.20	0.50 × 0.30 × 0.30
Data collection		
Diffractometer	Stoe IPDS2	Stoe IPDS2
Absorption correction	Multi-scan (<i>SORTAV</i> ; Blessing, 1995)	—
<i>T</i> _{min} , <i>T</i> _{max}	0.909, 0.963	—
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	2271, 1407, 806	4734, 3172, 1881
<i>R</i> _{int}	0.079	0.060
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.688	0.688
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.072, 0.193, 0.87	0.040, 0.085, 0.83
No. of reflections	1407	3172
No. of parameters	109	164
No. of restraints	12	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.30, -0.37	0.13, -0.17

Computer programs: *X-AREA* (Stoe & Cie, 2005), *SORTAV* (Blessing, 1987, 1989), *SHELXT* (Sheldrick, 2015a) and *SHELXL2014* (Sheldrick, 2015b).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.

There is no significant anomalous dispersion at this wavelength so the Flack parameter is meaningless and this is not reported.

For compound **1**, hydrogen atoms of the aromatic ring were placed using a riding model with the C—H bond length allowed to refine subject to the restraint that all these bond lengths were equal within a estimated standard deviation of 0.02 Å. These C—H bond lengths lie in the range 0.97 (3) to 0.99 (3) Å. The other hydrogen atoms attached to formally single-bonded nitrogen atoms were freely refined subject to sensible distance and angle restraints. The N—H distances lie in the range 0.94 (3)–0.95 (3) Å.

For compound **2**, hydrogen atoms were placed using a riding model [*N*—H = 0.88, C—H = 0.95–0.98 Å; *U*_{iso}(H) = 1.2 or 1.5*U*_{eq}(C)].

Acknowledgements

FEM thanks the Commonwealth Scholarship Commission in the United Kingdom for the generous Academic Fellowship CMCF-2015–3.

Funding information

Funding for this research was provided by: Commonwealth Scholarship Commission (award No. Academic Fellowship CMCF-2015-3).

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supporting information

Acta Cryst. (2017). E73, 1021-1025 [https://doi.org/10.1107/S2056989017008416]

Crystal structures of *N'*-aminopyridine-2-carboximidamide and *N'*-{[1-(pyridin-2-yl)ethylidene]amino}pyridine-2-carboximidamide

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Computing details

For both compounds, data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA* (Stoe & Cie, 2005). Data reduction: scaled and merged with *SORTAV* (Blessing, 1987, 1989) for (1); *X-AREA* (Stoe & Cie, 2005) for (2). For both compounds, program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b).

(1) *N'*-Aminopyridine-2-carboximidamide

Crystal data

$C_6H_8N_4$	$F(000) = 144$
$M_r = 136.16$	$D_x = 1.417 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 5.6955 (14) \text{ \AA}$	Cell parameters from 2199 reflections
$b = 3.8408 (5) \text{ \AA}$	$\theta = 5.5\text{--}28.6^\circ$
$c = 14.592 (4) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$\beta = 91.631 (19)^\circ$	$T = 150 \text{ K}$
$V = 319.08 (12) \text{ \AA}^3$	Block, colourless
$Z = 2$	$0.48 \times 0.21 \times 0.20 \text{ mm}$

Data collection

Stoe IPDS2	2271 measured reflections
diffractometer	1407 independent reflections
Radiation source: fine-focus sealed tube	806 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	$R_{\text{int}} = 0.079$
ω -scan	$\theta_{\text{max}} = 29.3^\circ$, $\theta_{\text{min}} = 2.8^\circ$
Absorption correction: multi-scan	$h = -7 \rightarrow 6$
(<i>SORTAV</i> ; Blessing, 1995)	$k = -4 \rightarrow 5$
$T_{\text{min}} = 0.909$, $T_{\text{max}} = 0.963$	$l = -20 \rightarrow 19$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.072$	$w = 1/[\sigma^2(F_o^2) + (0.1122P)^2]$
$wR(F^2) = 0.193$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.87$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1407 reflections	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
109 parameters	$\Delta\rho_{\text{min}} = -0.37 \text{ e \AA}^{-3}$
12 restraints	

Special details

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3776 (8)	0.3892 (13)	0.8928 (3)	0.0457 (11)
H1	0.494 (4)	0.4064 (14)	0.9427 (17)	0.055*
C2	0.1680 (8)	0.2331 (13)	0.9109 (3)	0.0464 (11)
H2	0.1358 (13)	0.143 (3)	0.973 (2)	0.056*
C3	0.0007 (9)	0.2040 (13)	0.8396 (3)	0.0462 (11)
H3	-0.152 (5)	0.092 (4)	0.8496 (4)	0.055*
C4	0.0545 (8)	0.3357 (12)	0.7545 (3)	0.0423 (11)
H4	-0.060 (4)	0.3176 (14)	0.7027 (17)	0.051*
C5	0.2711 (7)	0.4937 (12)	0.7426 (3)	0.0391 (9)
C6	0.3370 (7)	0.6479 (12)	0.6539 (3)	0.0369 (9)
N1	0.4337 (6)	0.5206 (10)	0.8109 (2)	0.0435 (10)
N2	0.5525 (7)	0.7974 (11)	0.6517 (2)	0.0443 (10)
N3	0.1844 (6)	0.6252 (9)	0.5862 (2)	0.0389 (9)
N4	0.2585 (6)	0.7713 (12)	0.5022 (2)	0.0422 (9)
H4A	0.125 (7)	0.840 (13)	0.466 (3)	0.063*
H2A	0.637 (8)	0.830 (16)	0.708 (2)	0.063*
H2B	0.614 (7)	0.954 (13)	0.608 (2)	0.063*
H4B	0.353 (7)	0.970 (12)	0.512 (3)	0.065*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.046 (3)	0.041 (3)	0.050 (2)	0.002 (2)	0.0008 (18)	0.000 (2)
C2	0.050 (3)	0.036 (3)	0.053 (2)	0.006 (2)	0.0041 (19)	0.0020 (19)
C3	0.042 (2)	0.037 (3)	0.060 (2)	0.003 (2)	0.0060 (18)	0.0037 (18)
C4	0.035 (2)	0.035 (3)	0.056 (2)	-0.001 (2)	0.0017 (18)	-0.0012 (19)
C5	0.035 (2)	0.031 (2)	0.051 (2)	0.007 (2)	-0.0021 (16)	-0.0032 (17)
C6	0.030 (2)	0.029 (2)	0.052 (2)	0.0006 (19)	0.0007 (16)	-0.0010 (17)
N1	0.041 (2)	0.037 (2)	0.0518 (19)	0.0003 (19)	0.0005 (14)	0.0001 (16)
N2	0.039 (2)	0.041 (2)	0.0528 (19)	-0.0051 (19)	0.0004 (15)	0.0007 (17)
N3	0.0346 (18)	0.033 (2)	0.0486 (18)	0.0019 (17)	0.0014 (14)	-0.0004 (16)
N4	0.041 (2)	0.038 (2)	0.0469 (17)	-0.0008 (19)	0.0006 (14)	0.0039 (16)

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

C1—N1	1.345 (6)	C5—N1	1.345 (5)
C1—C2	1.368 (7)	C5—C6	1.481 (5)
C1—H1	0.97 (3)	C6—N3	1.300 (5)
C2—C3	1.395 (7)	C6—N2	1.357 (6)

C2—H2	0.99 (3)	N2—H2A	0.95 (3)
C3—C4	1.383 (6)	N2—H2B	0.95 (3)
C3—H3	0.98 (3)	N3—N4	1.424 (5)
C4—C5	1.390 (6)	N4—H4A	0.95 (3)
C4—H4	0.99 (3)	N4—H4B	0.94 (3)
N1—C1—C2	124.5 (4)	N1—C5—C6	115.5 (4)
N1—C1—H1	117.8	C4—C5—C6	122.1 (4)
C2—C1—H1	117.8	N3—C6—N2	126.6 (4)
C1—C2—C3	118.2 (4)	N3—C6—C5	117.2 (4)
C1—C2—H2	120.9	N2—C6—C5	116.2 (3)
C3—C2—H2	120.9	C1—N1—C5	117.0 (4)
C4—C3—C2	118.4 (4)	C6—N2—H2A	118 (3)
C4—C3—H3	120.8	C6—N2—H2B	129 (2)
C2—C3—H3	120.8	H2A—N2—H2B	108 (3)
C3—C4—C5	119.5 (4)	C6—N3—N4	114.8 (4)
C3—C4—H4	120.3	N3—N4—H4A	110 (3)
C5—C4—H4	120.3	N3—N4—H4B	111 (3)
N1—C5—C4	122.4 (4)	H4A—N4—H4B	108 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2B \cdots N4 ⁱ	0.95 (3)	2.16 (3)	3.106 (5)	174 (4)
N4—H4B \cdots N4 ⁱ	0.94 (3)	2.51 (3)	3.357 (6)	149 (4)
N4—H4A \cdots N3 ⁱⁱ	0.95 (3)	2.19 (4)	3.113 (5)	162 (4)

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

(2) *N'*-[1-(Pyridin-2-yl)ethylidene]amino}pyridine-2-carboximidamide

Crystal data

C₁₃H₁₃N₅

M_r = 239.28

Orthorhombic, *P*2₁2₁2₁

a = 6.6899 (5) Å

b = 18.930 (2) Å

c = 9.6561 (11) Å

V = 1222.8 (2) Å³

Z = 4

F(000) = 504

D_x = 1.300 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 3070 reflections

θ = 2.2–27.9°

μ = 0.08 mm⁻¹

T = 150 K

Block, yellow

0.50 × 0.30 × 0.30 mm

Data collection

Stoe IPDS2

diffractometer

Radiation source: fine-focus sealed tube

Detector resolution: 6.67 pixels mm⁻¹

ω-scan

4734 measured reflections

3172 independent reflections

1881 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.060

θ_{max} = 29.3°, θ_{min} = 2.2°

h = -7→9

k = -22→25

l = -13→9

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.040$ $wR(F^2) = 0.085$ $S = 0.83$

3172 reflections

164 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0355P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.13 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$ *Special details*

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8155 (4)	0.19604 (14)	0.7310 (3)	0.0423 (6)
H1	0.8311	0.1496	0.7666	0.051*
C2	0.9604 (4)	0.22137 (15)	0.6439 (3)	0.0410 (6)
H2	1.0716	0.1929	0.6189	0.049*
C3	0.9409 (4)	0.28972 (15)	0.5928 (3)	0.0399 (6)
H3	1.0393	0.3092	0.5331	0.048*
C4	0.7756 (4)	0.32843 (13)	0.6309 (3)	0.0338 (6)
H4	0.7596	0.3754	0.5981	0.041*
C5	0.6314 (4)	0.29863 (12)	0.7179 (2)	0.0294 (5)
C6	0.4460 (4)	0.33685 (11)	0.7576 (2)	0.0292 (5)
C7	0.3185 (4)	0.30756 (13)	0.8722 (3)	0.0352 (6)
H7A	0.2303	0.3447	0.9076	0.053*
H7B	0.4045	0.2904	0.9472	0.053*
H7C	0.2377	0.2684	0.8365	0.053*
C8	0.1767 (4)	0.47107 (12)	0.6220 (3)	0.0289 (5)
C9	-0.0076 (4)	0.51400 (12)	0.6439 (2)	0.0287 (5)
C10	-0.1320 (4)	0.50366 (13)	0.7570 (3)	0.0350 (6)
H10	-0.1022	0.4685	0.8240	0.042*
C11	-0.3006 (4)	0.54571 (14)	0.7701 (3)	0.0405 (6)
H11	-0.3877	0.5401	0.8470	0.049*
C12	-0.3401 (4)	0.59572 (14)	0.6701 (3)	0.0390 (6)
H12	-0.4544	0.6253	0.6767	0.047*
C13	-0.2093 (4)	0.60183 (14)	0.5600 (3)	0.0382 (6)
H13	-0.2371	0.6365	0.4915	0.046*
N1	0.6516 (3)	0.23293 (11)	0.7696 (2)	0.0361 (5)
N2	0.4076 (3)	0.39235 (11)	0.6850 (2)	0.0312 (5)
N3	0.2326 (3)	0.42907 (10)	0.7220 (2)	0.0312 (5)
N4	0.2661 (3)	0.47642 (11)	0.4976 (2)	0.0396 (5)
H4A	0.3712	0.4503	0.4786	0.048*
H4B	0.2193	0.5061	0.4354	0.048*

N5	-0.0458 (3)	0.56182 (11)	0.5440 (2)	0.0344 (5)
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Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0446 (16)	0.0378 (13)	0.0444 (15)	0.0104 (13)	-0.0050 (13)	0.0018 (12)
C2	0.0358 (15)	0.0472 (15)	0.0399 (15)	0.0085 (13)	0.0000 (13)	-0.0054 (13)
C3	0.0345 (15)	0.0476 (16)	0.0376 (15)	-0.0026 (13)	0.0011 (12)	-0.0038 (12)
C4	0.0354 (14)	0.0309 (13)	0.0349 (14)	-0.0025 (11)	-0.0012 (12)	-0.0004 (11)
C5	0.0322 (13)	0.0266 (12)	0.0295 (12)	-0.0028 (11)	-0.0045 (11)	-0.0015 (10)
C6	0.0309 (13)	0.0251 (11)	0.0315 (13)	-0.0016 (10)	-0.0029 (11)	-0.0024 (10)
C7	0.0347 (14)	0.0327 (13)	0.0382 (14)	0.0020 (12)	0.0027 (12)	0.0026 (11)
C8	0.0290 (12)	0.0251 (12)	0.0326 (13)	-0.0034 (10)	0.0001 (11)	-0.0021 (10)
C9	0.0293 (12)	0.0258 (11)	0.0310 (12)	-0.0028 (10)	-0.0023 (10)	-0.0008 (10)
C10	0.0350 (13)	0.0334 (13)	0.0368 (14)	-0.0022 (11)	0.0006 (12)	0.0019 (11)
C11	0.0336 (14)	0.0454 (15)	0.0424 (14)	-0.0003 (12)	0.0072 (13)	-0.0018 (12)
C12	0.0354 (15)	0.0344 (14)	0.0472 (16)	0.0053 (12)	0.0009 (13)	-0.0013 (12)
C13	0.0397 (15)	0.0313 (13)	0.0437 (15)	0.0032 (13)	-0.0015 (12)	0.0036 (12)
N1	0.0361 (12)	0.0324 (11)	0.0398 (12)	0.0037 (10)	-0.0019 (10)	0.0045 (9)
N2	0.0327 (11)	0.0281 (11)	0.0326 (11)	0.0016 (10)	-0.0001 (9)	-0.0009 (9)
N3	0.0296 (11)	0.0301 (11)	0.0341 (11)	0.0013 (9)	0.0028 (9)	0.0009 (9)
N4	0.0389 (13)	0.0462 (13)	0.0338 (11)	0.0138 (11)	0.0048 (10)	0.0074 (10)
N5	0.0340 (12)	0.0299 (11)	0.0392 (12)	0.0025 (10)	-0.0003 (10)	0.0023 (9)

Geometric parameters (Å, °)

C1—N1	1.352 (3)	C8—N3	1.305 (3)
C1—C2	1.371 (4)	C8—N4	1.346 (3)
C1—H1	0.9500	C8—C9	1.492 (3)
C2—C3	1.391 (4)	C9—N5	1.347 (3)
C2—H2	0.9500	C9—C10	1.386 (4)
C3—C4	1.377 (4)	C10—C11	1.387 (3)
C3—H3	0.9500	C10—H10	0.9500
C4—C5	1.398 (3)	C11—C12	1.378 (4)
C4—H4	0.9500	C11—H11	0.9500
C5—N1	1.347 (3)	C12—C13	1.381 (4)
C5—C6	1.487 (3)	C12—H12	0.9500
C6—N2	1.289 (3)	C13—N5	1.340 (3)
C6—C7	1.503 (3)	C13—H13	0.9500
C7—H7A	0.9800	N2—N3	1.408 (3)
C7—H7B	0.9800	N4—H4A	0.8800
C7—H7C	0.9800	N4—H4B	0.8800
N1—C1—C2	124.2 (3)	N3—C8—C9	117.6 (2)
N1—C1—H1	117.9	N4—C8—C9	116.9 (2)
C2—C1—H1	117.9	N5—C9—C10	123.0 (2)
C1—C2—C3	118.5 (3)	N5—C9—C8	114.9 (2)
C1—C2—H2	120.8	C10—C9—C8	122.0 (2)

C3—C2—H2	120.8	C9—C10—C11	118.6 (2)
C4—C3—C2	118.4 (3)	C9—C10—H10	120.7
C4—C3—H3	120.8	C11—C10—H10	120.7
C2—C3—H3	120.8	C12—C11—C10	119.1 (3)
C3—C4—C5	120.0 (2)	C12—C11—H11	120.5
C3—C4—H4	120.0	C10—C11—H11	120.5
C5—C4—H4	120.0	C11—C12—C13	118.4 (3)
N1—C5—C4	121.7 (2)	C11—C12—H12	120.8
N1—C5—C6	116.0 (2)	C13—C12—H12	120.8
C4—C5—C6	122.3 (2)	N5—C13—C12	124.0 (2)
N2—C6—C5	115.0 (2)	N5—C13—H13	118.0
N2—C6—C7	126.0 (2)	C12—C13—H13	118.0
C5—C6—C7	118.9 (2)	C5—N1—C1	117.2 (2)
C6—C7—H7A	109.5	C6—N2—N3	115.5 (2)
C6—C7—H7B	109.5	C8—N3—N2	110.57 (19)
H7A—C7—H7B	109.5	C8—N4—H4A	120.0
C6—C7—H7C	109.5	C8—N4—H4B	120.0
H7A—C7—H7C	109.5	H4A—N4—H4B	120.0
H7B—C7—H7C	109.5	C13—N5—C9	116.9 (2)
N3—C8—N4	125.4 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N4—H4B \cdots N3 ⁱ	0.88	2.42	3.206 (3)	149

Symmetry code: (i) $-x+1/2, -y+1, z-1/2$.