

Pyridine-3-carbaldehyde 2-pyridyl-hydrazone

Lars Kr. Hansen,^{a*} German L. Perlovich,^{b,d} Klaus-Jürgen Schaper^c and Annette Bauer-Brandl^b

^aDepartment of Chemistry, University of Tromsø, 9037 Tromsø, Norway,

^bDepartment of Pharmaceutics and Biopharmaceutics, University of Tromsø, 9037 Tromsø, Norway, ^cResearch Center Borstel, Leibniz Center for Medicine and Biosciences, D-23845 Borstel, Germany, and ^dInstitute of Solution Chemistry, Russian Academy of Sciences, 153045 Ivanovo, Russian Federation
Correspondence e-mail: larsk@chem.uit.no

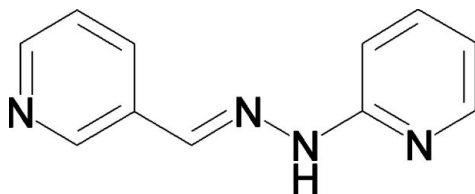
Received 28 March 2007; accepted 11 May 2007

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.045; wR factor = 0.047; data-to-parameter ratio = 20.4.

In the crystal structure of the title compound, $\text{C}_{11}\text{H}_{10}\text{N}_4$, molecules form dimers, which form a zigzag pattern in the crystal. The hydrogen-bond network can be described by graph-set notation as $R_2^2(14)$.

Related literature

For related literature, see: Allen *et al.* (1987); Cory *et al.* (1994); Etter (1990); Popp (1989); Seydel *et al.* (1994).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{10}\text{N}_4$
 $M_r = 198.23$
Monoclinic, $P2_1/n$
 $a = 10.896$ (5) Å
 $b = 4.0270$ (17) Å
 $c = 22.710$ (9) Å
 $\beta = 94.304$ (8)°

$V = 993.6$ (7) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293.1$ K
 $0.40 \times 0.30 \times 0.10$ mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan
Jacobson (1998)
 $T_{\min} = 0.892$, $T_{\max} = 0.992$

9025 measured reflections
3614 independent reflections
2273 reflections with $F^2 > 2\sigma(F^2)$
 $R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.048$
 $S = 0.97$
3614 reflections

177 parameters
All H-atom parameters refined
 $\Delta\rho_{\max} = 0.66$ e Å⁻³
 $\Delta\rho_{\min} = -0.52$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{N3}^i$	1.03 (2)	2.01 (2)	3.0371 (18)	177 (1)

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

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Molecular Structure Corporation & Rigaku, 2005); program(s) used to solve structure: *SIR88* (Burla *et al.*, 1989); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP III* (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

This study was supported by the Russian Foundation of Basic Research (No. 06-03-96304).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2161).

References

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supplementary materials

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Pyridine-3-carbaldehyde 2-pyridylhydrazone

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Comment

2-Pyridylhydrazone (2-pyridinylhydrazone 3-Pyridinecarboxaldehyde, I) belongs to the class of heterocyclic hydrazones, which have an anti-tuberculosis activity (Cory *et al.*, 1994; Seydel *et al.*, 1994). These compounds show some inhibitory activity towards ribonucleotide reductase and anticonvulsant activity as well (Popp, 1989). Crystal structure of (I) has not been solved before. Therefore the aim of the work was to fill this gap. A view of molecule (I) with the atomic numbering is presented in Fig 1. The parameters of the hydrogen bond geometry are shown in Table 1. The bond lengths are within the normal range of such bonds (Allen *et al.*, 1987). The conformational state of the molecule in the crystal structure can be characterized in the following way. The torsion angle N1—C1—C2—C3, which characterizes the orientation of the one pyridine fragment Py1 [N4—C6—C2—C3—C4—C5] with respect to bridge group (C1—N1—N2), is 3.5 (2) °. At other side, the torsion angle C8—C7—N2—N1, which describes the orientation of the second pyridine ring Py2 [N3—C7—C8—C9—C10—C11] with respect to the bridge group, is 0.9 (2) °. The torsion angle C2—C1—N1—N2, which characterizes a planarity of bridge group, is 178.86 (13) °. The pyridine fragments are rotated relatively to each other by 8.2 (2)°. The molecular packing architecture is shown in Figs. 2 & 3. The molecules of (I) form dimers by hydrogen bonds N2—H2...N3. The hydrogen-bond network can be described by the graph-set assignment introduced by Etter (1990) as $R_2^2(14)$. The dimers are packed in cups where interact to each other by van-der-Waals forces.

Experimental

The chemical synthesis of the title compound was performed by analogy to procedures described previously (Popp, 1989). Generally, the compound was recrystallized from methanol/water. The 2-Pyridylhydrazone crystal was grown by slow evaporation from methanol solution.

Figures

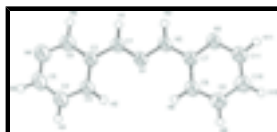


Fig. 1. A view of I with the atomic numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

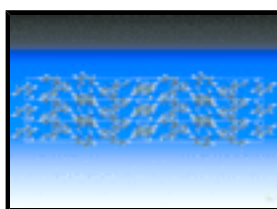


Fig. 2. Projection of crystal lattice molecular packing of (I) along OX axis.

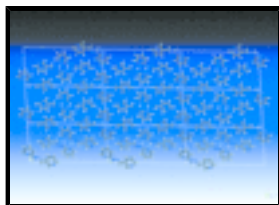


Fig. 3. Projection of crystal lattice molecular packing of (I) along OY axis.

Pyridine-3-carbaldehyde 2-pyridylhydrazone

Crystal data

$C_{11}H_{10}N_4$

$M_r = 198.23$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 10.896\ (5)\ \text{\AA}$

$b = 4.0270\ (17)\ \text{\AA}$

$c = 22.710\ (9)\ \text{\AA}$

$\beta = 94.304\ (8)^\circ$

$V = 993.6\ (7)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 416.00$

$D_x = 1.325\ \text{Mg m}^{-3}$

Melting point: 449 K

Mo $K\alpha$ radiation

$\lambda = 0.71070\ \text{\AA}$

Cell parameters from 1851 reflections

$\theta = 3.2\text{--}30.0^\circ$

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

$T = 293.1\ \text{K}$

Prism, colorless

$0.40 \times 0.30 \times 0.10\ \text{mm}$

Data collection

Rigaku Saturn
diffractometer

ω scans

Absorption correction: multi-scan
Jacobson (1998)

$T_{\min} = 0.892$, $T_{\max} = 0.992$

9025 measured reflections

3614 independent reflections

2273 reflections with $F^2 > 2\sigma(F^2)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 30.4^\circ$

$h = -14 \rightarrow 14$

$k = -3 \rightarrow 5$

$l = -32 \rightarrow 31$

Standard reflections: ?;

every ? reflections

intensity decay: ?

Refinement

Refinement on F

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.048$

$S = 0.97$

3614 reflections

177 parameters

All H-atom parameters refined

Chebyshev polynomial with 3 parameters (Carruthers & Watkin, 1979) 88.1628 106.9210 44.1569

$(\Delta/\sigma)_{\max} = 0.006$

$\Delta\rho_{\max} = 0.66\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.52\ \text{e \AA}^{-3}$

Extinction correction: Larson (1970) Crystallographic Computing eq. 22

Extinction coefficient: 41 (21)

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement using reflections with $F^2 > 3.0 \text{ sigma}(F^2)$. The weighted R -factor(wR), goodness of fit (S) and R -factor (gt) are based on F , with F set to zero for negative F . The threshold expression of $F^2 > 2.0 \text{ sigma}(F^2)$ is used only for calculating R -factor (gt).

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.58600 (12)	0.1829 (3)	0.62487 (5)	0.0552 (4)
N2	0.57422 (12)	0.2776 (3)	0.56706 (5)	0.0583 (4)
N3	0.64083 (11)	0.2790 (3)	0.47412 (5)	0.0573 (4)
N4	0.39775 (16)	0.1814 (4)	0.80823 (6)	0.0817 (6)
C1	0.49701 (17)	0.2595 (4)	0.65614 (6)	0.0565 (5)
C2	0.50216 (14)	0.1730 (4)	0.71847 (6)	0.0508 (5)
C3	0.60286 (18)	0.0237 (4)	0.74843 (6)	0.0572 (5)
C4	0.6002 (2)	-0.0391 (4)	0.80754 (8)	0.0679 (6)
C5	0.4975 (2)	0.0415 (5)	0.83536 (9)	0.0752 (7)
C6	0.40282 (18)	0.2461 (5)	0.75060 (8)	0.0685 (6)
C7	0.66146 (14)	0.1757 (4)	0.53006 (6)	0.0501 (5)
C8	0.76185 (16)	-0.0184 (4)	0.54956 (8)	0.0579 (5)
C9	0.84375 (19)	-0.1077 (4)	0.50947 (9)	0.0680 (6)
C10	0.82507 (18)	-0.0028 (4)	0.45175 (8)	0.0669 (6)
C11	0.72443 (17)	0.1861 (4)	0.43666 (8)	0.0614 (6)
H1	0.4229 (13)	0.386 (3)	0.6392 (5)	0.059 (4)*
H2	0.4999 (17)	0.419 (4)	0.5523 (7)	0.101 (6)*
H3	0.6766 (12)	-0.036 (3)	0.7269 (5)	0.058 (4)*
H4	0.6726 (14)	-0.133 (4)	0.8288 (6)	0.075 (5)*
H5	0.4973 (14)	0.002 (4)	0.8809 (7)	0.092 (5)*
H6	0.3308 (14)	0.349 (3)	0.7279 (6)	0.078 (5)*
H8	0.7715 (12)	-0.100 (3)	0.5907 (6)	0.061 (4)*
H9	0.9192 (14)	-0.251 (4)	0.5233 (6)	0.078 (5)*
H10	0.8840 (13)	-0.067 (3)	0.4231 (6)	0.069 (4)*
H11	0.7058 (13)	0.271 (3)	0.3930 (6)	0.071 (4)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0598 (9)	0.0669 (10)	0.0390 (7)	-0.0061 (7)	0.0043 (6)	0.0036 (6)
N2	0.0522 (9)	0.0841 (11)	0.0390 (7)	0.0041 (8)	0.0049 (7)	0.0107 (7)
N3	0.0558 (8)	0.0752 (10)	0.0411 (7)	-0.0023 (7)	0.0056 (6)	0.0025 (7)
N4	0.0849 (12)	0.1059 (13)	0.0575 (10)	-0.0055 (10)	0.0267 (9)	0.0024 (9)
C1	0.0503 (11)	0.0746 (14)	0.0447 (10)	0.0025 (10)	0.0046 (8)	0.0070 (9)
C2	0.0500 (10)	0.0583 (11)	0.0445 (8)	-0.0014 (8)	0.0070 (8)	0.0002 (8)
C3	0.0602 (11)	0.0648 (12)	0.0472 (10)	-0.0018 (10)	0.0072 (9)	0.0035 (9)
C4	0.0756 (14)	0.0746 (13)	0.0529 (12)	-0.0008 (11)	-0.0003 (11)	0.0077 (10)

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C5	0.0966 (17)	0.0823 (15)	0.0478 (11)	-0.0209 (13)	0.0121 (12)	0.0046 (11)
C6	0.0600 (12)	0.0914 (16)	0.0552 (11)	0.0032 (11)	0.0129 (10)	0.0052 (11)
C7	0.0486 (10)	0.0576 (11)	0.0445 (9)	-0.0085 (9)	0.0056 (8)	-0.0009 (8)
C8	0.0608 (12)	0.0626 (12)	0.0499 (10)	-0.0022 (9)	0.0026 (9)	0.0042 (10)
C9	0.0669 (13)	0.0690 (14)	0.0688 (13)	0.0071 (10)	0.0104 (10)	0.0033 (10)
C10	0.0723 (14)	0.0683 (13)	0.0626 (11)	0.0054 (11)	0.0224 (10)	-0.0035 (10)
C11	0.0668 (12)	0.0700 (13)	0.0483 (10)	-0.0022 (10)	0.0107 (9)	-0.0009 (9)

Geometric parameters (Å, °)

N1—N2	1.3640 (16)	C8—C9	1.370 (2)
N1—C1	1.282 (2)	C9—C10	1.378 (2)
N2—C7	1.378 (2)	C10—C11	1.357 (2)
N3—C7	1.3396 (19)	N2—H2	1.026 (18)
N3—C11	1.345 (2)	C1—H1	1.007 (14)
N4—C5	1.334 (2)	C3—H3	1.001 (14)
N4—C6	1.340 (2)	C4—H4	0.971 (15)
C1—C2	1.455 (2)	C5—H5	1.048 (17)
C2—C3	1.384 (2)	C6—H6	0.996 (15)
C2—C6	1.382 (2)	C8—H8	0.988 (14)
C3—C4	1.368 (2)	C9—H9	1.034 (16)
C4—C5	1.364 (3)	C10—H10	0.981 (15)
C7—C8	1.390 (2)	C11—H11	1.054 (14)
N2—N1—C1	116.45 (13)	C7—N2—H2	122.0 (9)
N1—N2—C7	118.97 (13)	N1—C1—H1	122.0 (8)
C7—N3—C11	116.11 (13)	C2—C1—H1	117.6 (8)
C5—N4—C6	116.20 (18)	C2—C3—H3	120.0 (7)
N1—C1—C2	120.39 (15)	C4—C3—H3	120.8 (7)
C1—C2—C3	123.61 (15)	C3—C4—H4	118.9 (9)
C1—C2—C6	119.24 (15)	C5—C4—H4	121.8 (9)
C3—C2—C6	117.15 (15)	N4—C5—H5	117.3 (8)
C2—C3—C4	119.20 (17)	C4—C5—H5	119.0 (8)
C3—C4—C5	119.28 (19)	N4—C6—H6	119.8 (9)
N4—C5—C4	123.69 (18)	C2—C6—H6	115.7 (9)
N4—C6—C2	124.47 (17)	C7—C8—H8	120.8 (8)
N2—C7—N3	114.31 (13)	C9—C8—H8	120.9 (8)
N2—C7—C8	122.40 (14)	C8—C9—H9	119.2 (8)
N3—C7—C8	123.29 (15)	C10—C9—H9	121.1 (8)
C7—C8—C9	118.19 (16)	C9—C10—H10	119.5 (8)
C8—C9—C10	119.65 (18)	C11—C10—H10	122.4 (8)
C9—C10—C11	118.10 (18)	N3—C11—H11	114.5 (8)
N3—C11—C10	124.66 (16)	C10—C11—H11	120.9 (8)
N1—N2—H2	119.0 (9)		
N2—N1—C1—C2	-179.10 (14)	C1—C2—C3—C4	178.10 (17)
C1—N1—N2—C7	-174.09 (15)	C1—C2—C6—N4	-179.27 (17)
N1—N2—C7—N3	179.33 (13)	C3—C2—C6—N4	-0.2 (2)
N1—N2—C7—C8	-0.5 (2)	C6—C2—C3—C4	-1.0 (2)
C7—N3—C11—C10	0.5 (2)	C2—C3—C4—C5	1.2 (2)
C11—N3—C7—N2	179.64 (14)	C3—C4—C5—N4	-0.2 (3)

C11—N3—C7—C8	-0.5 (2)	N2—C7—C8—C9	179.95 (14)
C5—N4—C6—C2	1.1 (2)	N3—C7—C8—C9	0.1 (2)
C6—N4—C5—C4	-0.8 (3)	C7—C8—C9—C10	0.4 (2)
N1—C1—C2—C3	3.7 (2)	C8—C9—C10—C11	-0.4 (2)
N1—C1—C2—C6	-177.31 (16)	C9—C10—C11—N3	-0.1 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 \cdots N3 ⁱ	1.026 (18)	2.012 (18)	3.0371 (18)	176.5 (13)

Symmetry codes: (i) $-x+1, -y+1, -z+1$.

Fig. 1

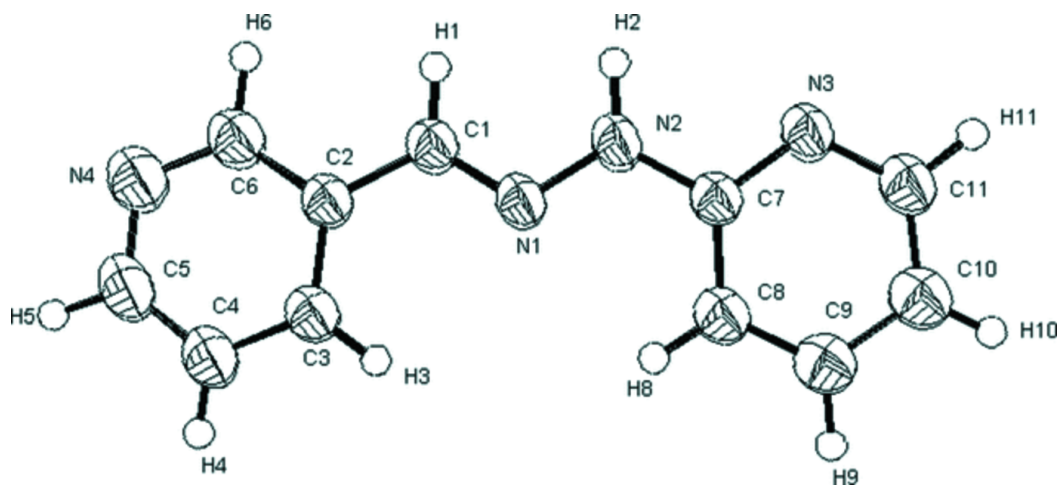


Fig. 2

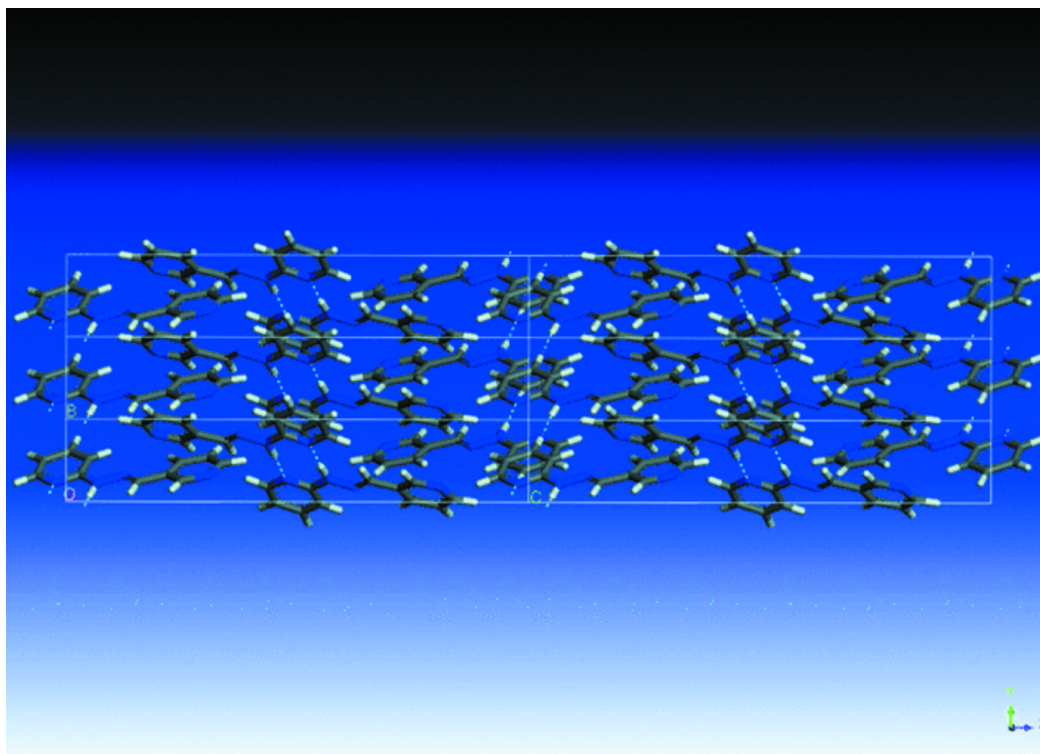


Fig. 3

