Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-Hydroxypyridine-4-carboxamide

Qingkun Wu and Handong Yin*

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China
Correspondence e-mail: handongyin@163.com

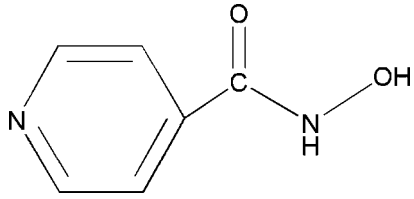
Received 2 June 2011; accepted 29 June 2011

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.115; data-to-parameter ratio = 11.9.

The title compound, $C_6H_6N_2O_2$, is approximately planar with an r.m.s. deviation for the non-H atoms of 0.052 Å. In the crystal, a two-dimensional array in the *bc* plane is stabilized by O—H···N and N—H···O hydrogen bonds.

Related literature

For background to the coordination chemistry of hydroxamic acid derivatives, see: Codd (2008). For related structures, see: Wang *et al.* (1988); Makhmudova *et al.* (2000); Golenya *et al.* (2007).



Experimental

Crystal data

$C_6H_6N_2O_2$
M_r = 138.13
Monoclinic, $P2_1/c$
a = 4.8765 (5) Å
b = 13.4476 (16) Å
c = 9.6656 (11) Å
β = 99.579 (1)°
V = 625.01 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 K
0.35 × 0.24 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.961, T_{max} = 0.983
3030 measured reflections
1092 independent reflections
769 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.115
S = 1.05
1092 reflections
92 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2^{i}	0.82	1.92	2.721 (2)	166
N1—H2···O2^{ii}	0.86	2.01	2.844 (2)	162

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We acknowledge the National Natural Science Foundation of China (20771053), the National Basic Research Program (No. 2010CB234601) and the Natural Science Foundation of Shandong Province (Y2008B48) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2754).

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supporting information

Acta Cryst. (2011). E**67**, o1905 [doi:10.1107/S1600536811025566]

N-Hydroxypyridine-4-carboxamide

Qingkun Wu and Handong Yin

S1. Comment

Hydroxamic acid, R1C(═O)N(R2)OH (R1 = alkyl/aryl; R2 = alkyl/aryl or H), relevant to chemical biology, coordinate a wide variety of metal ions predominantly as the monoanionic hydroxamato or as a dianionic (R2 = H) hydroximato *O*,*O*-bidentate chelate (Codd, 2008). To the best of our knowledge, while a large number of transition metal derivatives with hydroxamic acids have been reported, organoantimony complexes with hydroxamic acids are limited. To further extend this field and to construct novel structures of organoantimony, we choose to investigate reactions involving the title compound (I). Herein, we present its crystal structure determination.

In (I), the non-hydrogen atoms are in the same plane (Fig. 1) with the rms deviation being 0.052 Å. All the bond lengths and angles are normal and correspond to those observed in the related compounds (Wang *et al.*, 1988; Makhmudova *et al.*, 2000; Golenya *et al.*, 2007). In the crystal structure, intermolecular O1—H1···N2 and N1—H2···O2 hydrogen bonds (Table 1) link the molecules into a two-dimensional array in the *bc* plane (Fig. 2).

S2. Experimental

4-pyridinecarboxylic acid was dissolved in methanol (50 mL) and concentrated sulfuric acid (5 mL) was added drop wise into the reactor. The mixture was then stirred and refluxed for 3 h, after which time the solution was adjusted to pH 8 by the use of a 5% sodium carbonate aqueous solution. Methyl 4-pyridinecarboxylate was obtained by extraction with diethyl ether. Yield: 61.6%. A mixture of hydroxylamine hydrochloride and sodium hydroxide was added drop wise to the methanol solution of methyl 4-pyridinecarboxylate. The reaction was allowed to continue at room temperature for 72 h, when the mixture was acidified to pH 5.5 by 5% HCl solution. The solvent was removed *in vacuo* and the product was recrystallized from water to give pale-red crystals. Yield: 82%, M.pt. 421 K.

S3. Refinement

The H atoms were geometrically placed (O—H = 0.82 Å, N—H = 0.86 Å and C—H = 0.93 Å) and refined as riding with U_{iso}(H) = 1.2-1.5U_{eq}(parent atom).

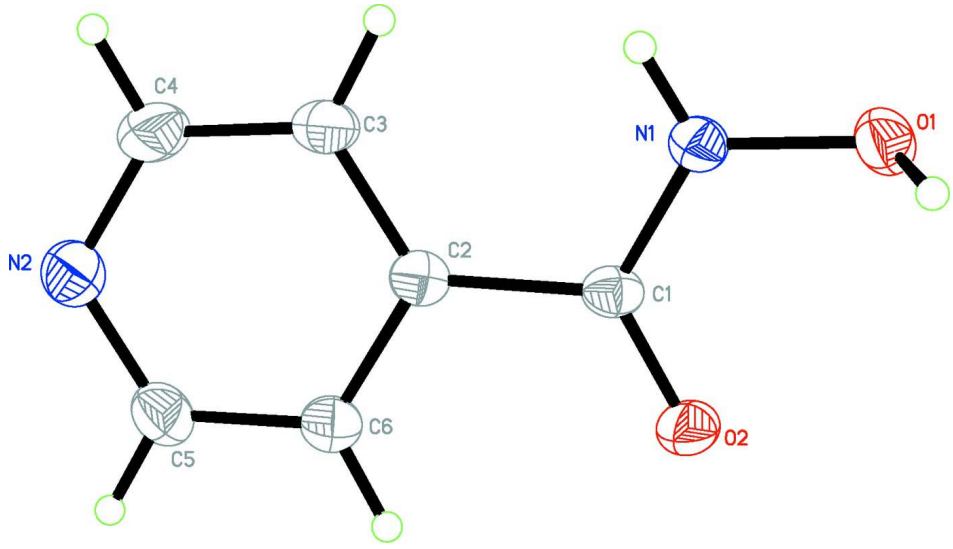

Figure 1

The molecular structure of (I), showing atom-labelling and 50% probability displacement ellipsoids.

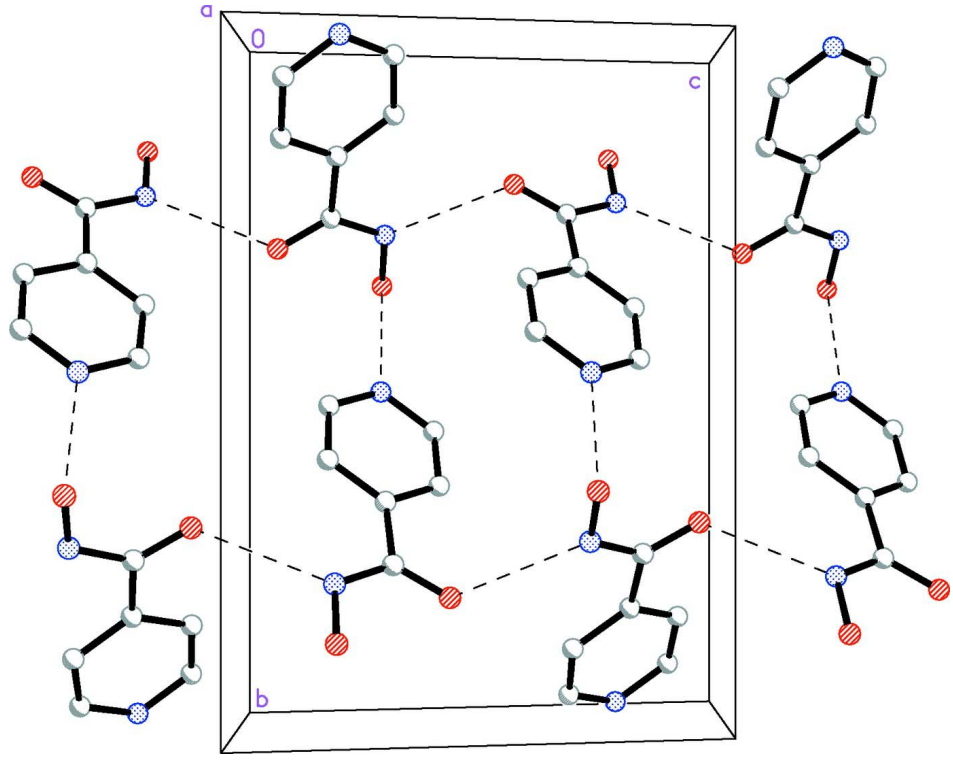

Figure 2

The supramolecular 2-D array in the bc plane stabilised by N—H···O and O—H···N hydrogen bonds (dashed lines).

N-Hydroxypyridine-4-carboxamide

Crystal data

$C_6H_6N_2O_2$
M_r = 138.13

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

a = 4.8765 (5) Å
b = 13.4476 (16) Å
c = 9.6656 (11) Å
β = 99.579 (1)°
V = 625.01 (12) Å^3
Z = 4
F(000) = 288
D_x = 1.468 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1066 reflections
θ = 2.6–26.0°
μ = 0.11 mm^{-1}
T = 298 K
Block, pale-red
0.35 × 0.24 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.961, T_{max} = 0.983

3030 measured reflections
1092 independent reflections
769 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.0°, θ_{min} = 2.6°
h = −5→5
k = −13→16
l = −11→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.115
S = 1.05
1092 reflections
92 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
O2	0.1487 (3)	0.30086 (10)	0.06585 (14)	0.0430 (4)
O1	−0.1208 (3)	0.35086 (10)	0.27794 (16)	0.0437 (5)
H1	−0.0465	0.4055	0.2770	0.065*
N1	0.0809 (4)	0.27699 (12)	0.28861 (17)	0.0364 (5)
H2	0.1275	0.2453	0.3662	0.044*
C1	0.2019 (4)	0.25531 (14)	0.1785 (2)	0.0324 (5)
C2	0.4074 (4)	0.17092 (14)	0.1980 (2)	0.0331 (5)
N2	0.7903 (4)	0.01429 (13)	0.2167 (2)	0.0477 (5)
C6	0.5567 (5)	0.15118 (16)	0.0916 (2)	0.0420 (6)
H6	0.5325	0.1905	0.0114	0.050*
C3	0.4594 (6)	0.11050 (18)	0.3153 (2)	0.0570 (7)
H3	0.3672	0.1214	0.3908	0.068*
C5	0.7415 (5)	0.07300 (17)	0.1049 (2)	0.0462 (6)
H5	0.8377	0.0606	0.0313	0.055*
C4	0.6486 (6)	0.03405 (19)	0.3196 (3)	0.0659 (8)
H4	0.6790	−0.0061	0.3990	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0581 (10)	0.0425 (9)	0.0303 (8)	0.0067 (8)	0.0127 (7)	0.0035 (6)
O1	0.0467 (9)	0.0354 (8)	0.0525 (10)	0.0019 (7)	0.0187 (7)	−0.0029 (7)
N1	0.0442 (11)	0.0354 (10)	0.0312 (9)	0.0040 (8)	0.0107 (8)	−0.0002 (7)
C1	0.0404 (12)	0.0305 (10)	0.0272 (10)	−0.0053 (9)	0.0084 (9)	−0.0039 (9)
C2	0.0382 (12)	0.0292 (10)	0.0325 (11)	−0.0044 (9)	0.0078 (9)	−0.0043 (8)
N2	0.0514 (12)	0.0356 (10)	0.0584 (12)	0.0025 (9)	0.0162 (10)	0.0023 (9)
C6	0.0448 (13)	0.0485 (14)	0.0341 (12)	0.0047 (11)	0.0103 (10)	0.0022 (9)
C3	0.0782 (19)	0.0518 (15)	0.0495 (14)	0.0206 (13)	0.0353 (13)	0.0163 (11)
C5	0.0455 (14)	0.0488 (14)	0.0469 (14)	0.0045 (11)	0.0156 (11)	−0.0046 (11)
C4	0.090 (2)	0.0537 (16)	0.0620 (17)	0.0249 (15)	0.0364 (16)	0.0262 (13)

Geometric parameters (Å, º)

O2—C1	1.239 (2)	N2—C5	1.327 (3)
O1—N1	1.390 (2)	N2—C4	1.329 (3)
O1—H1	0.8200	C6—C5	1.377 (3)
N1—C1	1.332 (2)	C6—H6	0.9300
N1—H2	0.8600	C3—C4	1.378 (3)
C1—C2	1.505 (3)	C3—H3	0.9300
C2—C6	1.381 (3)	C5—H5	0.9300
C2—C3	1.384 (3)	C4—H4	0.9300
N1—O1—H1	109.5	C5—C6—H6	120.2
C1—N1—O1	119.96 (16)	C2—C6—H6	120.2
C1—N1—H2	120.0	C4—C3—C2	119.5 (2)
O1—N1—H2	120.0	C4—C3—H3	120.2
O2—C1—N1	122.57 (19)	C2—C3—H3	120.2
O2—C1—C2	121.35 (17)	N2—C5—C6	123.8 (2)
N1—C1—C2	116.07 (17)	N2—C5—H5	118.1
C6—C2—C3	116.7 (2)	C6—C5—H5	118.1
C6—C2—C1	118.33 (18)	N2—C4—C3	123.8 (2)
C3—C2—C1	124.92 (18)	N2—C4—H4	118.1
C5—N2—C4	116.4 (2)	C3—C4—H4	118.1
C5—C6—C2	119.7 (2)		
O1—N1—C1—O2	2.4 (3)	C1—C2—C6—C5	−178.48 (19)
O1—N1—C1—C2	−177.12 (15)	C6—C2—C3—C4	−1.3 (4)
O2—C1—C2—C6	6.1 (3)	C1—C2—C3—C4	178.6 (2)
N1—C1—C2—C6	−174.36 (18)	C4—N2—C5—C6	0.2 (3)
O2—C1—C2—C3	−173.7 (2)	C2—C6—C5—N2	−0.9 (3)
N1—C1—C2—C3	5.8 (3)	C5—N2—C4—C3	−0.1 (4)
C3—C2—C6—C5	1.4 (3)	C2—C3—C4—N2	0.6 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2^{i}	0.82	1.92	2.721 (2)	166
N1—H2···O2^{ii}	0.86	2.01	2.844 (2)	162

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