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N′-Hydroxynicotinamidine

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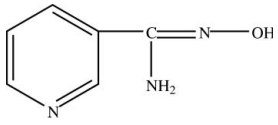
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.060; wR factor = 0.158; data-to-parameter ratio = 14.0.

In the title compound, $C_6H_7N_3O$, there are inter- and intramolecular N—H···N hydrogen bonds and an O—H···N hydrogen bond.

Related literature

For related literature, see: Gezginci *et al.* (2001).



Experimental

Crystal data

$C_6H_7N_3O$
M_r = 137.15
Monoclinic, $P2_1/c$
a = 5.5220 (11) Å
b = 12.365 (3) Å
c = 9.797 (2) Å
β = 94.38 (3)°
V = 667.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.951, T_{max} = 0.970
1444 measured reflections
1307 independent reflections
782 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.158
S = 1.16
1307 reflections
93 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···N3^{i}	0.98	2.11	3.023 (4)	155
O1—H1A···N1^{ii}	0.82	1.95	2.767 (4)	176
N2—H2A···O1	0.99	2.13	2.539 (4)	103

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x+1, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXS97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2034).

References

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supplementary materials

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N'-Hydroxynicotinamidine

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Comment

Some derivatives of nicotinamidine is important chemical material. We report here the crystal structure of the title compound, (I).

The molecular structure of (I) is shown in Fig. 1,

Experimental

Nicotinonitrile(20 mmol) was dissolved in ethanol (8 ml), hydroxylamine hydrochloride(20 mmol) was dissolved in ethanol (6 ml) and potassium carbonate (10 mmol) was dissolved in water (10 ml). The three separate solutions were mixed and the resulting mixture was refluxed for 24 h. After cooling and filtrating, the crude compound (I) was obtained. Pure compound (I) was obtained by crystallizing from a mixture of ethanol (6 ml) and water (2 ml). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 8.98 (s, 1H), 8.74 (m, 1H), 8.18 (m, 1H), 7.57 (m, 1H), 2.36 (s, 3H).

Refinement

All H atoms bonded to the C atoms were placed geometrically at the distances of 0.93–0.97 Å and included in the refinement in riding motion approximation with U_{iso}(H) = 1.2 or 1.5U_{eq} of the carrier atom. The H atoms attached to N2 were located on a difference map and refined as isotropically asriding atoms. The H atom attached to O1 was refined as a riding atom at a distance of 0.82Å from the O1 with Uĩso~(H) = 1.2U~eq~ of O1.

Figures

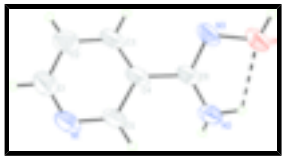

Fig. 1. A view of the molecular structure of (I), showing displacement ellipsoids at the 30% probability level. Dashed lines indicate N—H···N hydrogen bonds and O—H···N hydrogen bond

N'-Hydroxynicotinamidine

Crystal data

$C_6H_7N_3O$	F_{000} = 288
M_r = 137.15	D_x = 1.366 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å

Hall symbol: -p 2ybc	Cell parameters from 25 reflections
a = 5.5220 (11) Å	θ = 9–13º
b = 12.365 (3) Å	μ = 0.10 mm^{-1}
c = 9.797 (2) Å	T = 293 (2) K
β = 94.38 (3)º	Block, colourless
V = 667.0 (3) $Å^3$	0.20 × 0.20 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.053
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 2.7º
T = 293(2) K	h = −6→6
ω/2θ scans	k = 0→15
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→12
T_{min} = 0.951, T_{max} = 0.970	3 standard reflections
1444 measured reflections	every 200 reflections
1307 independent reflections	intensity decay: none
782 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.060	H-atom parameters constrained
$wR(F^2)$ = 0.158	$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.3602P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.16	$(\Delta/\sigma)_{max}$ = 0.002
1307 reflections	$\Delta\rho_{max}$ = 0.21 e $Å^{-3}$
93 parameters	$\Delta\rho_{min}$ = −0.22 e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.6266 (4)	0.85703 (19)	0.2247 (2)	0.0575 (7)
H1A	0.6800	0.8630	0.1492	0.086*
N1	−0.1856 (5)	0.6347 (2)	0.4713 (3)	0.0523 (7)
N2	0.4172 (5)	0.8367 (2)	0.4451 (3)	0.0538 (8)
H2A	0.5697	0.8776	0.4355	0.072 (11)*
H2B	0.4664	0.8136	0.5387	0.111 (16)*
N3	0.4391 (5)	0.7778 (2)	0.2184 (3)	0.0466 (7)
C1	−0.2182 (6)	0.5548 (3)	0.3844 (4)	0.0564 (9)
H1B	−0.3460	0.5073	0.3953	0.068*
C2	−0.0749 (6)	0.5366 (3)	0.2776 (4)	0.0568 (9)
H2C	−0.1067	0.4794	0.2172	0.068*
C3	0.1172 (6)	0.6057 (3)	0.2633 (3)	0.0501 (9)
H3B	0.2206	0.5942	0.1941	0.060*
C4	0.1560 (6)	0.6916 (2)	0.3512 (3)	0.0424 (8)
C5	0.0006 (6)	0.7027 (3)	0.4547 (3)	0.0468 (8)
H5A	0.0259	0.7599	0.5158	0.056*
C6	0.3564 (5)	0.7710 (2)	0.3388 (3)	0.0406 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0685 (16)	0.0605 (15)	0.0481 (13)	−0.0083 (13)	0.0346 (11)	0.0048 (12)
N1	0.0523 (17)	0.0572 (17)	0.0510 (16)	0.0048 (15)	0.0278 (13)	0.0007 (15)
N2	0.071 (2)	0.0523 (17)	0.0416 (16)	−0.0005 (15)	0.0278 (14)	0.0007 (14)
N3	0.0516 (16)	0.0485 (16)	0.0433 (15)	0.0003 (13)	0.0275 (12)	0.0037 (12)
C1	0.055 (2)	0.058 (2)	0.059 (2)	−0.0029 (18)	0.0205 (18)	0.0033 (18)
C2	0.073 (2)	0.0444 (19)	0.056 (2)	−0.0005 (19)	0.0236 (18)	−0.0053 (17)
C3	0.062 (2)	0.052 (2)	0.0397 (17)	0.0036 (18)	0.0284 (16)	0.0020 (15)
C4	0.0523 (19)	0.0395 (17)	0.0384 (16)	0.0085 (15)	0.0235 (14)	0.0056 (13)
C5	0.0506 (18)	0.0514 (19)	0.0417 (17)	0.0002 (17)	0.0247 (15)	−0.0041 (15)
C6	0.0460 (18)	0.0427 (18)	0.0355 (16)	0.0118 (14)	0.0194 (13)	0.0068 (14)

Geometric parameters (Å, °)

O1—N3	1.423 (3)	C1—H1B	0.9300
O1—H1A	0.8200	C2—C3	1.378 (4)
N1—C1	1.308 (4)	C2—H2C	0.9300
N1—C5	1.348 (4)	C3—C4	1.373 (4)
N2—C6	1.343 (4)	C3—H3B	0.9300
N2—H2A	0.9929	C4—C5	1.385 (4)
N2—H2B	0.9787	C4—C6	1.491 (4)
N3—C6	1.300 (4)	C5—H5A	0.9300
C1—C2	1.378 (4)		
N3—O1—H1A	109.5	C4—C3—C2	120.0 (3)

C1—N1—C5	117.5 (3)	C4—C3—H3B	120.0
C6—N2—H2A	113.5	C2—C3—H3B	120.0
C6—N2—H2B	125.8	C3—C4—C5	117.4 (3)
H2A—N2—H2B	93.5	C3—C4—C6	122.6 (3)
C6—N3—O1	108.1 (2)	C5—C4—C6	119.9 (3)
N1—C1—C2	124.0 (3)	N1—C5—C4	123.2 (3)
N1—C1—H1B	118.0	N1—C5—H5A	118.4
C2—C1—H1B	118.0	C4—C5—H5A	118.4
C3—C2—C1	117.9 (3)	N3—C6—N2	125.5 (3)
C3—C2—H2C	121.1	N3—C6—C4	115.5 (3)
C1—C2—H2C	121.1	N2—C6—C4	118.6 (2)
C5—N1—C1—C2	−0.2 (5)	C6—C4—C5—N1	179.3 (3)
N1—C1—C2—C3	1.3 (5)	O1—N3—C6—N2	7.4 (4)
C1—C2—C3—C4	−2.1 (5)	O1—N3—C6—C4	179.7 (2)
C2—C3—C4—C5	1.9 (5)	C3—C4—C6—N3	22.5 (4)
C2—C3—C4—C6	−178.3 (3)	C5—C4—C6—N3	−157.7 (3)
C1—N1—C5—C4	0.0 (5)	C3—C4—C6—N2	−164.7 (3)
C3—C4—C5—N1	−0.9 (5)	C5—C4—C6—N2	15.1 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2B···N3^{i}	0.98	2.11	3.023 (4)	155
O1—H1A···N1^{ii}	0.82	1.95	2.767 (4)	176
N2—H2A···O1	0.99	2.13	2.539 (4)	103

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) *x*+1, −*y*+3/2, *z*−1/2.

Fig. 1