Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(2-Nitrooxyethyl)picolinamide

Drielly A. Paixão,[a] Silvana Guilardi,[a]* Ângelo de Fátima,[b] Débora P. Araujo[b] and Francinely C. Oliveira[b]

[a]Instituto de Química – UFU, Uberlândia, MG, Brazil, and [b]Departamento de Química – UFMG, Belo Horizonte, MG, Brazil
Correspondence e-mail: silvana@ufu.br

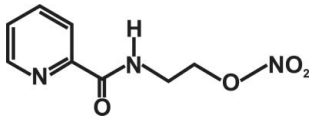
Received 15 September 2011; accepted 26 September 2011

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.108; data-to-parameter ratio = 9.2.

In the title molecule, $C_8H_9N_3O_4$, the amide group is involved in the formation of an intramolecular N—H···N hydrogen bond. In the crystal, molecules related by translation along the a axis are linked into chains *via* weak intermolecular C—H···O interactions.

Related literature

For related structures, see: Eremenko *et al.* (1996); Fedorov *et al.* (2001). For further synthetic details, see: Samejima (1960); Jiao *et al.* (1990).



Experimental

Crystal data

$C_8H_9N_3O_4$
M_r = 211.18
Orthorhombic, $P2_12_12_1$
a = 5.5075 (2) Å
b = 13.6114 (5) Å
c = 12.6822 (4) Å
V = 950.72 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 295 K
0.49 × 0.21 × 0.19 mm

Data collection

Nonius KappaCCD diffractometer
4037 measured reflections
1265 independent reflections
1039 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.108
S = 1.07
1265 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H···N1	0.86	2.31	2.692 (3)	107
C8—H8B···O1^{i}	0.97	2.39	3.239 (3)	145

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank Professor Dr Javier Ellena of the IFSC, USP, Brazil, for the X-ray data collection. This work was supported financially by FAPEMIG and CNPq.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5154).

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supporting information

Acta Cryst. (2011). E**67**, o2816 [https://doi.org/10.1107/S1600536811039572]

N-(2-Nitrooxyethyl)picolinamide

Drielly A. Paixão, Silvana Guilardi, Ângelo de Fátima, Débora P. Araujo and Francinely C. Oliveira

S1. Comment

The title compound (I) can be considered as a potential nitric oxide donating drug. Herewith we present its crystal structure.

The molecule of (I) adopts a folded conformation and contains a planar pyridine cycle (NC_5H_4) bearing a CO group attached to the α-carbon atom (Fig. 1). The dihedral angle between the pyridine ring and the O1/C6/N2 plane is 11.3 (2)°. This angle is smaller than that in the nicorandyl [22.8 (2)°] (Eremenko *et al.*, 1996).The dihedral angle between the mean planes O1/C6/N2/C7 and C8/O2/N3/O3/O4 is 44.74 (7)°. The C═O and CH_2–ONO_2 bonds are oriented *trans* to the pyridine nitrogen atom. In the nicorandyl compound these groups were found in the *cis* position (Eremenko *et al.*, 1996). Another structural isomer (Fedorov *et al.*, 2001), [*N*-(2-nitrooxyethyl)isonicotinamide], displays a different molecular conformation and crystallizes in the same centrosymmetric space group $P2_1/c$ as nicorandil.

The crystal structure of (I) is stabilized through weak non-classical intermolecular H-bonds of the type C—H···O in [100] direction, involving the carbon atom of the nitrooxyethyl group and the oxygen atom of carbonylamide. Moreover, were observed one intramolecular interactions of the type N—H···N (Table 1). On the other hand, the compound nicorandil has only one intermolecular interaction of the type N—H···O. The results for compound (I) and its structural isomers show that the position of the ligand in pyridine ring affects the conformation of the molecule and the interactions present in the crystal packing.

S2. Experimental

The title product was synthesized by heating ethylnicotinate with an excess 2-ethanolamine to give *N*-(2-hydroxyethyl)-picolinamide in 92% yield (Samejima, 1960). The nitration of *N*-(2-hydroxyethyl)picolinamide (0.10 mmol) was held by mixing it with fuming nitric acid (1.00 mmol) at -5°C and stirred for 2 h.

The reaction mixture was poured into water and ice, and the pH was adjusted to 6.0 adding ($CaCO_3$). The white solid obtained was filtered at reduced pressure and recrystallized in ethanol, forming the *N*-(2-nitrooxyethyl)picolinamide in 63% yield (Jiao *et al.*, 1990). MP: 61.2–63.0°C. ^{1}H-NMR (200 MHz, $CDCl_3$): 3.85 (2*H*, q, J = 5.6 Hz), 4.67 (2*H*, t, J = 5.2 Hz),7.42–7.48 (^{1}H, m), 7.83–7.90 (^{1}H, m), 8.19 (^{1}H, d, J = 7.9 Hz), 8.39 (^{1}H, br s), 8.56 (^{1}H, d, J = 4.4 Hz). ^{13}C-NMR (200 MHz, $CDCl_3$): 36.7, 71.7, 122.2, 126.4, 137.4, 148.1, 149.11, 164.7.

S3. Refinement

H atoms were geometrically positioned (C—H 0.93-0.97 Å, N—H 0.86 Å) and refined as riding, with U_{iso}(H) = 1.2 U_{eq} of the parent atom. In the absence of significant anomalous scatterers in the molecule, attempts to confirm the absolute structure by refinement of the Flack parameter in the presence of 871 sets of Friedel equivalents led to an inconclusive value of -0.2 (13). Therefore, the Friedel pairs were merged before the final refinement.

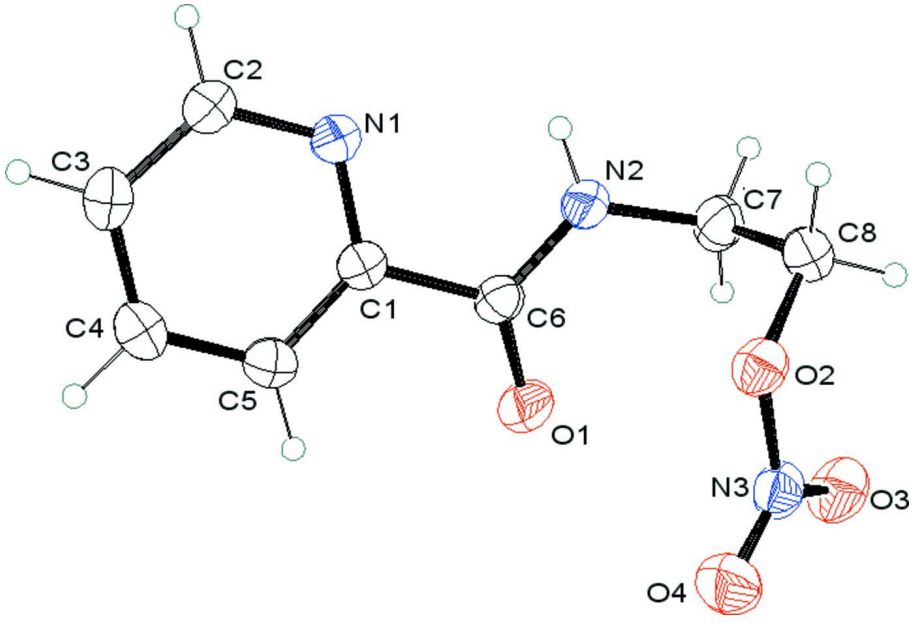

Figure 1

The molecular structure of (I) showing the atom labeling. Displacement ellipsoids are drawn at the 30% probability level.

N-(2-Nitrooxyethyl)pyridine-2-carboxamide

Crystal data

$C_8H_9N_3O_4$
$M_r = 211.18$
Orthorhombic, $P2_12_12_1$
$a = 5.5075$ (2) Å
$b = 13.6114$ (5) Å
$c = 12.6822$ (4) Å
$V = 950.72$ (6) Å^3
$Z = 4$
$F(000) = 440$
$D_x = 1.475$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2165 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.12$ mm^{-1}
$T = 295$ K
Prism, colourless
0.49 × 0.21 × 0.19 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Enraf–Nonius
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
4037 measured reflections
1265 independent reflections
1039 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -7 \rightarrow 7$
$k = -16 \rightarrow 17$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.108$
$S = 1.07$
1265 reflections
137 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
$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2 + 0.0645P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Extinction correction: *SHELXL*
Extinction coefficient: 0.124 (13)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.1820 (3)	0.49141 (13)	0.05722 (14)	0.0662 (5)
O2	0.2098 (3)	0.55263 (11)	−0.11459 (12)	0.0528 (4)
O3	−0.0499 (4)	0.67762 (12)	−0.11167 (15)	0.0705 (5)
O4	−0.1235 (4)	0.54631 (16)	−0.20249 (14)	0.0797 (6)
N1	0.1892 (3)	0.31135 (13)	0.17680 (14)	0.0509 (5)
N2	0.2136 (4)	0.49676 (13)	0.10379 (15)	0.0532 (5)
H	0.3331	0.4648	0.1308	0.064*
N3	−0.0061 (4)	0.59684 (13)	−0.14445 (15)	0.0532 (5)
C1	−0.0042 (4)	0.34497 (13)	0.12528 (15)	0.0430 (5)
C2	0.1867 (5)	0.21623 (15)	0.20374 (19)	0.0570 (6)
H2	0.3191	0.1914	0.2407	0.068*
C3	−0.0002 (5)	0.15319 (15)	0.17994 (18)	0.0565 (6)
H3	0.0066	0.0875	0.1998	0.068*
C4	−0.1974 (5)	0.18928 (16)	0.12612 (19)	0.0561 (6)
H4	−0.3265	0.1484	0.1086	0.067*
C5	−0.2003 (4)	0.28722 (15)	0.09855 (18)	0.0516 (5)
H5	−0.332	0.3138	0.0626	0.062*
C6	−0.0002 (4)	0.45146 (14)	0.09262 (15)	0.0459 (5)
C7	0.2509 (5)	0.59775 (15)	0.07192 (18)	0.0592 (6)
H7A	0.3618	0.629	0.1209	0.071*
H7B	0.0973	0.6325	0.0754	0.071*
C8	0.3520 (5)	0.60631 (17)	−0.03806 (19)	0.0595 (6)
H8A	0.3569	0.6751	−0.0581	0.071*
H8B	0.5171	0.5816	−0.0387	0.071*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0545 (10)	0.0624 (10)	0.0817 (12)	0.0089 (8)	−0.0073 (9)	0.0174 (9)
O2	0.0556 (9)	0.0472 (7)	0.0555 (8)	0.0094 (7)	0.0037 (7)	0.0021 (6)
O3	0.0751 (12)	0.0563 (9)	0.0800 (12)	0.0189 (9)	0.0063 (10)	0.0087 (9)
O4	0.0799 (13)	0.0921 (13)	0.0672 (11)	−0.0228 (11)	−0.0137 (10)	0.0037 (10)

N1	0.0505 (10)	0.0471 (9)	0.0550 (10)	−0.0004 (8)	−0.0099 (9)	0.0039 (8)
N2	0.0585 (12)	0.0429 (8)	0.0581 (11)	−0.0035 (8)	−0.0129 (9)	0.0069 (8)
N3	0.0538 (11)	0.0526 (10)	0.0533 (11)	0.0014 (9)	0.0050 (9)	0.0093 (8)
C1	0.0447 (11)	0.0457 (10)	0.0385 (10)	0.0022 (9)	0.0016 (9)	−0.0010 (7)
C2	0.0584 (13)	0.0492 (11)	0.0633 (14)	0.0021 (10)	−0.0085 (12)	0.0074 (10)
C3	0.0685 (15)	0.0433 (10)	0.0577 (13)	−0.0026 (11)	0.0050 (12)	0.0021 (9)
C4	0.0574 (13)	0.0521 (12)	0.0588 (12)	−0.0105 (11)	0.0019 (11)	−0.0068 (10)
C5	0.0455 (11)	0.0559 (12)	0.0534 (12)	−0.0002 (9)	−0.0029 (10)	−0.0011 (9)
C6	0.0491 (12)	0.0459 (9)	0.0428 (10)	0.0022 (9)	−0.0026 (9)	0.0003 (8)
C7	0.0773 (17)	0.0433 (10)	0.0570 (13)	−0.0099 (11)	−0.0131 (12)	0.0011 (9)
C8	0.0519 (13)	0.0536 (12)	0.0728 (16)	−0.0090 (10)	−0.0059 (12)	0.0097 (11)

Geometric parameters (Å, º)

O1—C6	1.225 (3)	C2—C3	1.374 (3)
O2—N3	1.385 (3)	C2—H2	0.93
O2—C8	1.445 (3)	C3—C4	1.374 (4)
O3—N3	1.200 (2)	C3—H3	0.93
O4—N3	1.197 (3)	C4—C5	1.378 (3)
N1—C1	1.330 (3)	C4—H4	0.93
N1—C2	1.339 (3)	C5—H5	0.93
N2—C6	1.337 (3)	C7—C8	1.506 (3)
N2—C7	1.447 (3)	C7—H7A	0.97
N2—H	0.86	C7—H7B	0.97
C1—C5	1.378 (3)	C8—H8A	0.97
C1—C6	1.508 (3)	C8—H8B	0.97
N3—O2—C8	115.41 (16)	C5—C4—H4	120.6
C1—N1—C2	116.73 (19)	C4—C5—C1	118.7 (2)
C6—N2—C7	122.2 (2)	C4—C5—H5	120.7
C6—N2—H	118.9	C1—C5—H5	120.7
C7—N2—H	118.9	O1—C6—N2	123.66 (18)
O4—N3—O3	129.1 (2)	O1—C6—C1	121.03 (18)
O4—N3—O2	112.47 (19)	N2—C6—C1	115.30 (18)
O3—N3—O2	118.4 (2)	N2—C7—C8	112.60 (19)
N1—C1—C5	123.50 (19)	N2—C7—H7A	109.1
N1—C1—C6	116.99 (18)	C8—C7—H7A	109.1
C5—C1—C6	119.49 (19)	N2—C7—H7B	109.1
N1—C2—C3	123.7 (2)	C8—C7—H7B	109.1
N1—C2—H2	118.1	H7A—C7—H7B	107.8
C3—C2—H2	118.1	O2—C8—C7	112.49 (19)
C4—C3—C2	118.6 (2)	O2—C8—H8A	109.1
C4—C3—H3	120.7	C7—C8—H8A	109.1
C2—C3—H3	120.7	O2—C8—H8B	109.1
C3—C4—C5	118.8 (2)	C7—C8—H8B	109.1
C3—C4—H4	120.6	H8A—C8—H8B	107.8
C8—O2—N3—O4	−175.41 (19)	C7—N2—C6—O1	−1.7 (3)

C8—O2—N3—O3	5.3 (3)	C7—N2—C6—C1	177.33 (19)
C2—N1—C1—C5	−0.5 (3)	N1—C1—C6—O1	−170.5 (2)
C2—N1—C1—C6	−178.70 (19)	C5—C1—C6—O1	11.2 (3)
C1—N1—C2—C3	0.9 (3)	N1—C1—C6—N2	10.4 (3)
N1—C2—C3—C4	−0.5 (4)	C5—C1—C6—N2	−167.8 (2)
C2—C3—C4—C5	−0.3 (3)	C6—N2—C7—C8	−94.3 (3)
C3—C4—C5—C1	0.6 (3)	N3—O2—C8—C7	76.5 (2)
N1—C1—C5—C4	−0.2 (3)	N2—C7—C8—O2	52.7 (3)
C6—C1—C5—C4	177.95 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H···N1	0.86	2.31	2.692 (3)	107
C8—H8*B*···O1^{i}	0.97	2.39	3.239 (3)	145

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