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Nicotinium nitrate monohydrate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.137
Data-to-parameter ratio = 11.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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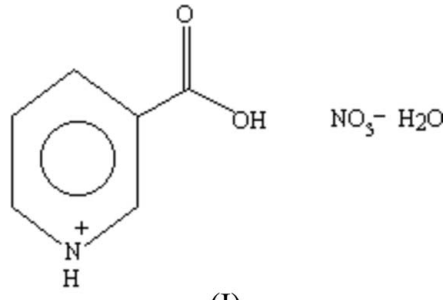
In the title compound, $C_6H_6NO_2^+\cdot NO_3^-\cdot H_2O$, the nicotinium cation is essentially planar. N—H···O, O—H···O and C—H···O hydrogen bonds link the molecules into layers parallel to the $(10\bar{1})$ plane.

Comment

Nicotinic acid (vitamin B3), known as niacin, is a lipid lowering agent widely used to treat hypertriglyceridemia by the inhibition of lipolysis in adipose tissue (Athimoolam & Rajaram, 2005). The nicotinic acid complex 5-methylpyrazine-2-carboxylic acid-4-oxide is a commonly used drug for the treatment of hypercholesterolemia (Lorenzen *et al.*, 2001). Coordination complexes of nicotinic acid with metals such as Sn possess antitumour activity greater than the well known *cis*-platin or doxorubicin (Gielen *et al.*, 1992). The enzyme nicotinic acid mononucleotide adenyltransferase is essential for the synthesis of nicotinamide adenine dinucleotide in all living cells and is a potential target for antibiotics (Kim *et al.*, 2004). As a part of our investigation of inorganic salts of nicotinic acid, we report here the crystal structure of nicotinium nitrate monohydrate, (I).



(I)

The asymetric unit of (I) contains a nicotinium cation, a nitrate anion and a water molecule (Fig. 1). Protonation of atom N1 of nicotine results in a widening of the C2—N1—C6 angle to 122.9 (3)°, compared with 118.9 (3)° in unprotonated nicotinic acid (Kutoglu & Scheringer, 1983). The nicotinium cation is essentially planar, with a maximum deviation from the mean plane of 0.048 (2) Å for atom O1.

The crystal packing is stabilized by N—H···O, O—H···O and C—H···O hydrogen bonds (Table 1), which link the molecules into layers parallel to the $(10\bar{1})$ plane (Fig. 2).

Experimental

Nitric acid was added dropwise to an aqueous solution of nicotinic acid, in stoichiometric amounts. The solution was heated at 323 K for

2 h. Colourless block-shaped crystals of (I) were obtained by slow evaporation over a period of one week.

Crystal data

$C_6H_6NO_2^+\cdot NO_3^-\cdot H_2O$
$M_r = 204.14$
Monoclinic, $P2_1/n$
$a = 6.6539\ (7)$ Å
$b = 12.3682\ (15)$ Å
$c = 10.1814\ (15)$ Å
$\beta = 100.967\ (7)^\circ$
$V = 822.59\ (18)$ Å^3
$Z = 4$
$D_x = 1.641$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.15$ mm^{-1}
$T = 120\ (2)$ K
Block, colourless
0.2 × 0.2 × 0.07 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.970$, $T_{max} = 0.990$
6153 measured reflections
1604 independent reflections
894 reflections with $I > 2\sigma(I)$
$R_{int} = 0.109$
$\theta_{max} = 26.1^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.137$
$S = 0.98$
1604 reflections
135 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	1.93	2.782 (3)	170
O1—H1A···O6	0.82	1.77	2.587 (3)	180
O6—H6A···O5	0.93 (5)	1.92 (5)	2.843 (3)	171 (4)
O6—H6B···O3^{ii}	0.88 (5)	1.96 (5)	2.825 (3)	173 (5)
C2—H2···O2^{iii}	0.93	2.43	3.173 (4)	137
C4—H4···O1^{iv}	0.93	2.46	3.262 (4)	144
C6—H6···O5^{i}	0.93	2.35	3.051 (4)	132
C6—H6···O4^{iv}	0.93	2.32	3.013 (4)	131

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

Water H atoms were located in a difference map and refined freely [O—H = 0.88 (5) and 0.93 (5) Å]. All other H atoms were placed in calculated positions, with C—H = 0.93 Å, O—H = 0.82 Å and N—H = 0.86 Å, and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C,N)$ and $1.5U_{eq}(O)$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

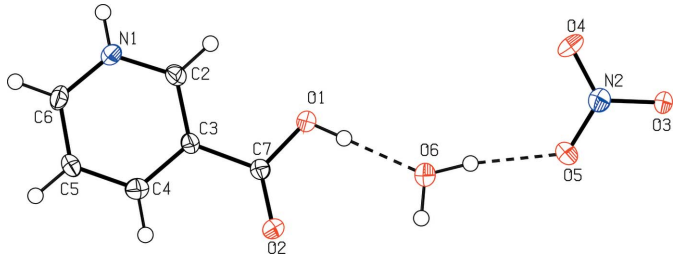


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme, with 50% probability displacement ellipsoids. Hydrogen bonds are drawn as dashed lines.

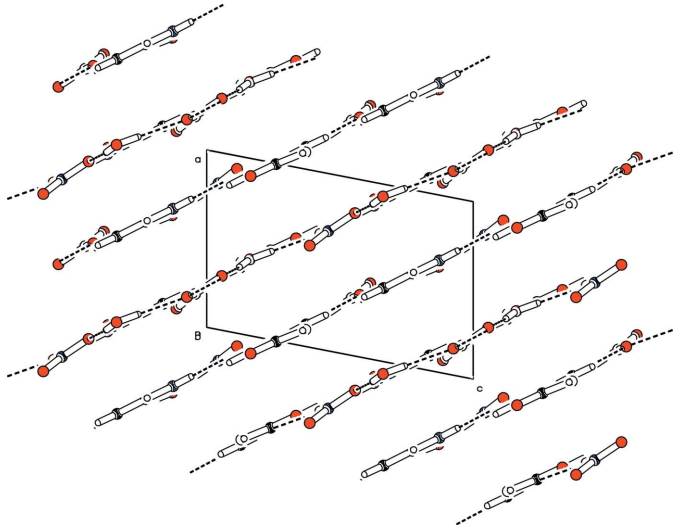


Figure 2
A packing diagram for (I), viewed down the *b* axis. Hydrogen bonds are drawn as dashed lines.

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supporting information

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Hall symbol: -P 2yn
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$b = 12.3682$ (15) Å
$c = 10.1814$ (15) Å
$\beta = 100.967$ (7)°
$V = 822.59$ (18) Å^3
$Z = 4$
$F(000) = 424$
$D_x = 1.641$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 1.0$–$26.0°$
$\mu = 0.15$ mm^{-1}
$T = 120$ K
Block, colourless
0.2 × 0.2 × 0.07 mm

Data collection

Bruker–Nonius FR591 rotating anode diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.970$, $T_{max} = 0.990$
6153 measured reflections
1604 independent reflections
894 reflections with $I > 2\sigma(I)$
$R_{int} = 0.109$
$\theta_{max} = 26.1°$, $\theta_{min} = 3.3°$
$h = -8 \rightarrow 8$
$k = -14 \rightarrow 14$
$l = -12 \rightarrow 9$
3 standard reflections every 60 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.137$
$S = 0.98$
1604 reflections
135 parameters
0 restraints
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.0945 (3)	0.36210 (16)	0.3701 (2)	0.0259 (6)
H1A	0.0866	0.4279	0.3602	0.039*
O2	−0.0994 (3)	0.36163 (16)	0.1648 (2)	0.0258 (6)
N1	0.1318 (3)	0.03157 (19)	0.3733 (3)	0.0205 (7)
H1	0.2044	−0.001	0.4406	0.025*
C2	0.1259 (4)	0.1401 (2)	0.3736 (3)	0.0196 (7)
H2	0.1984	0.1786	0.4458	0.023*
C3	0.0112 (4)	0.1940 (2)	0.2659 (3)	0.0167 (7)
C4	−0.0953 (4)	0.1334 (2)	0.1597 (3)	0.0211 (8)
H4	−0.1732	0.168	0.0861	0.025*
C5	−0.0851 (4)	0.0216 (2)	0.1637 (3)	0.0219 (8)
H5	−0.1556	−0.0191	0.0929	0.026*
C6	0.0292 (4)	−0.0283 (2)	0.2724 (3)	0.0209 (8)
H6	0.0361	−0.1034	0.2764	0.025*
C7	−0.0042 (4)	0.3138 (2)	0.2603 (3)	0.0181 (7)
N2	0.3245 (4)	0.8028 (2)	0.5438 (3)	0.0235 (7)
O3	0.3502 (3)	0.90208 (16)	0.5744 (2)	0.0243 (6)
O4	0.4266 (3)	0.73309 (17)	0.6135 (2)	0.0342 (7)
O5	0.1940 (3)	0.77703 (17)	0.4435 (2)	0.0268 (6)
O6	0.0695 (4)	0.56951 (18)	0.3383 (3)	0.0264 (6)
H6A	0.124 (6)	0.634 (4)	0.377 (4)	0.067 (14)*
H6B	0.002 (6)	0.584 (4)	0.258 (5)	0.082 (17)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0344 (12)	0.0150 (12)	0.0236 (14)	0.0002 (10)	−0.0062 (10)	−0.0002 (10)
O2	0.0352 (12)	0.0176 (12)	0.0210 (13)	0.0032 (10)	−0.0040 (10)	0.0015 (10)
N1	0.0219 (14)	0.0155 (14)	0.0227 (16)	0.0005 (11)	0.0003 (11)	0.0030 (12)
C2	0.0169 (16)	0.0196 (17)	0.0207 (18)	−0.0013 (13)	−0.0002 (13)	−0.0022 (14)
C3	0.0182 (16)	0.0153 (17)	0.0163 (18)	−0.0003 (12)	0.0023 (13)	−0.0026 (13)
C4	0.0198 (16)	0.0225 (18)	0.0204 (19)	0.0011 (13)	0.0021 (13)	0.0017 (15)
C5	0.0252 (17)	0.0186 (18)	0.0200 (18)	−0.0050 (14)	−0.0008 (13)	−0.0035 (14)
C6	0.0237 (17)	0.0125 (16)	0.0265 (19)	−0.0012 (13)	0.0048 (14)	0.0011 (14)
C7	0.0187 (16)	0.0168 (16)	0.0179 (18)	−0.0021 (13)	0.0015 (14)	0.0003 (14)
N2	0.0266 (15)	0.0199 (16)	0.0235 (17)	−0.0015 (12)	0.0037 (13)	0.0010 (13)
O3	0.0339 (12)	0.0131 (12)	0.0227 (13)	−0.0011 (10)	−0.0027 (9)	0.0000 (10)
O4	0.0420 (14)	0.0201 (13)	0.0351 (16)	0.0088 (11)	−0.0065 (12)	0.0080 (11)
O5	0.0318 (13)	0.0226 (12)	0.0225 (14)	−0.0040 (10)	−0.0039 (11)	−0.0027 (10)
O6	0.0303 (13)	0.0179 (13)	0.0276 (15)	−0.0025 (10)	−0.0027 (11)	0.0000 (11)

Geometric parameters (Å, º)

O1—C7	1.326 (3)	C4—C5	1.384 (4)
O1—H1A	0.82	C4—H4	0.93

O2—C7	1.209 (3)	C5—C6	1.365 (4)
N1—C2	1.342 (4)	C5—H5	0.93
N1—C6	1.343 (4)	C6—H6	0.93
N1—H1	0.86	N2—O4	1.235 (3)
C2—C3	1.383 (4)	N2—O5	1.249 (3)
C2—H2	0.93	N2—O3	1.271 (3)
C3—C4	1.393 (4)	O6—H6A	0.93 (5)
C3—C7	1.486 (4)	O6—H6B	0.88 (5)
C7—O1—H1A	109.5	C6—C5—C4	119.5 (3)
C2—N1—C6	122.9 (3)	C6—C5—H5	120.2
C2—N1—H1	118.6	C4—C5—H5	120.2
C6—N1—H1	118.6	N1—C6—C5	119.6 (3)
N1—C2—C3	119.4 (3)	N1—C6—H6	120.2
N1—C2—H2	120.3	C5—C6—H6	120.2
C3—C2—H2	120.3	O2—C7—O1	123.9 (3)
C2—C3—C4	118.6 (3)	O2—C7—C3	122.7 (3)
C2—C3—C7	122.3 (3)	O1—C7—C3	113.4 (2)
C4—C3—C7	119.0 (3)	O4—N2—O5	120.8 (2)
C5—C4—C3	119.9 (3)	O4—N2—O3	120.0 (2)
C5—C4—H4	120	O5—N2—O3	119.2 (2)
C3—C4—H4	120	H6A—O6—H6B	108 (4)
C6—N1—C2—C3	−0.5 (5)	C2—N1—C6—C5	0.8 (5)
N1—C2—C3—C4	0.0 (5)	C4—C5—C6—N1	−0.6 (5)
N1—C2—C3—C7	179.3 (3)	C2—C3—C7—O2	177.8 (3)
C2—C3—C4—C5	0.1 (5)	C4—C3—C7—O2	−2.9 (5)
C7—C3—C4—C5	−179.2 (3)	C2—C3—C7—O1	−2.8 (4)
C3—C4—C5—C6	0.2 (5)	C4—C3—C7—O1	176.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.86	1.93	2.782 (3)	170
O1—H1*A*···O6	0.82	1.77	2.587 (3)	180
O6—H6*A*···O5	0.93 (5)	1.92 (5)	2.843 (3)	171 (4)
O6—H6*B*···O3^{ii}	0.88 (5)	1.96 (5)	2.825 (3)	173 (5)
C2—H2···O2^{iii}	0.93	2.43	3.173 (4)	137
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Symmetry codes: (i) *x*, *y*−1, *z*; (ii) *x*−1/2, −*y*+3/2, *z*−1/2; (iii) *x*+1/2, −*y*+1/2, *z*+1/2; (iv) *x*−1/2, −*y*+1/2, *z*−1/2.