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3-Carboxypyridinium nitrate

Khalid Al-Farhan, Miftahul Khair and Mohamed Ghazzali*

Department of Chemistry, College of Science, King Saud University, PO Box 2455, Riyadh 11451, Saudi Arabia
Correspondence e-mail: mghazzali@ksu.edu.sa

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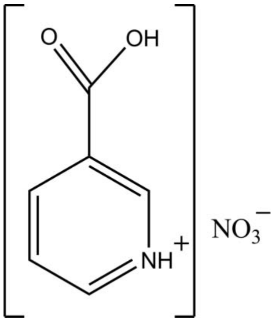
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.145; data-to-parameter ratio = 13.9.

In the crystal structure of the title compound, $C_6H_6NO_2^+\cdot NO_3^-$, the protonated cations are linked by N—H···O hydrogen bonds into chains along the *b* axis. The cations and anions are also linked by N—H···O and O—H···O hydrogen bonds. C—H···O interactions also occur. In the cation, the ring makes a dihedral angle of 10.1 (3)° with the carboxylate group.

Related literature

For related structures, see: Athimoolam & Rajaram (2005); Athimoolam & Natarajan (2007); Kutoglu & Scheringer (1983); Jebas *et al.* (2006); Slouf (2001); Ye *et al.* (2010). For graph-set descriptors, see: Etter (1990); Bernstein *et al.* (1995); Motherwell *et al.* (2000).



Experimental

Crystal data

$C_6H_6NO_2^+\cdot NO_3^-$
M_r = 186.13
Triclinic, $P\bar{1}$
a = 6.7530 (4) Å
b = 7.5024 (4) Å
c = 8.4439 (5) Å
α = 81.895 (2)°
β = 82.215 (1)°
γ = 66.769 (2)°
V = 387.69 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 K
0.40 × 0.20 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)
T_{min} = 0.946, T_{max} = 0.986
15468 measured reflections
1760 independent reflections
1102 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.145
S = 1.13
1760 reflections
127 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···O4^{i}	0.92 (3)	1.67 (3)	2.5833 (19)	169 (2)
N1—H1*N*···O2^{ii}	0.96 (3)	2.08 (3)	2.824 (2)	133 (2)
N1—H1*N*···O4	0.96 (3)	2.12 (3)	2.921 (2)	139 (2)
C3—H3···O3	0.93	2.45	3.330 (3)	158
C6—H6···O5^{iii}	0.93	2.37	3.259 (2)	160
C5—H5···O3^{iv}	0.93	2.47	3.142 (2)	129

Symmetry codes: (i) $x, y-1, z$; (ii) $x, y+1, z$; (iii) $x, y-1, z+1$; (iv) $x, y, z+1$.

Data collection: *CrystalClear* (Rigaku, 2007); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2004); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2240).

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supplementary materials

Acta Cryst. (2012). E**68**, o2246 [doi:10.1107/S1600536812028565]

3-Carboxypyridinium nitrate

Khalid Al-Farhan, Miftahul Khair and Mohamed Ghazzali

Comment

Previously, the crystal structures of nicotinium derivatives containing diverse anions have been reported (Athimoolam & Rajaram, 2005; Athimoolam & Natarajan, 2007; Kutoglu & Scheringer, 1983; Jebas *et al.*, 2006; Slouf, 2001; Ye *et al.*, 2010). We report herein the crystal structure of the title compound.

In the title molecule (Fig. 1), the nicotinium cation is planar with a maximum deviation for the carboxylate oxygen atom (O1) being 0.265 (2) Å. In the crystal structure (Fig. 2), the cations are linked by N—H···O hydrogen bonds (Table 1) into infinite chains along the [010] vector. Regarding the graph set descriptors (Etter, 1990; Bernstein *et al.*, 1995; Motherwell *et al.*, 2000), this N—H···O chain motif is described as C(6). The bifurcated N—H···O with O—H···O interactions (Table 1) are connecting the nicotinium with nitrates, thus defining a third-level discrete $D^3_3(13)$ hydrogen bond motif in the *bc*-plane (Fig. 2).

Experimental

The title compound was unintentionally obtained during a microwave irradiation (300 W, 150 ^{o}C, 10 min., MicroSynth, Milestone) reaction of $Ce(OH)_4$ in diluted nitric acid with aqueous solution of nicotinic acid. After cooling, the solution was left undisturbed and colourless crystals were collected by filtration after one week.

Refinement

Aromatic carbon–bound H–atoms were placed in ideal calculated positions [C—H 0.93 Å, $U_{iso}(H) = 1.2U_{eq}(C)$] and refined as riding atoms. Amine and hydroxyl hydrogen atoms were located from difference Fourier map and refined freely.

Computing details

Data collection: *CrystalClear* (Rigaku, 2007); cell refinement: *CrystalClear* (Rigaku, 2007); data reduction: *CrystalClear* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2004); software used to prepare material for publication: *publCIF* (Westrip, 2010).

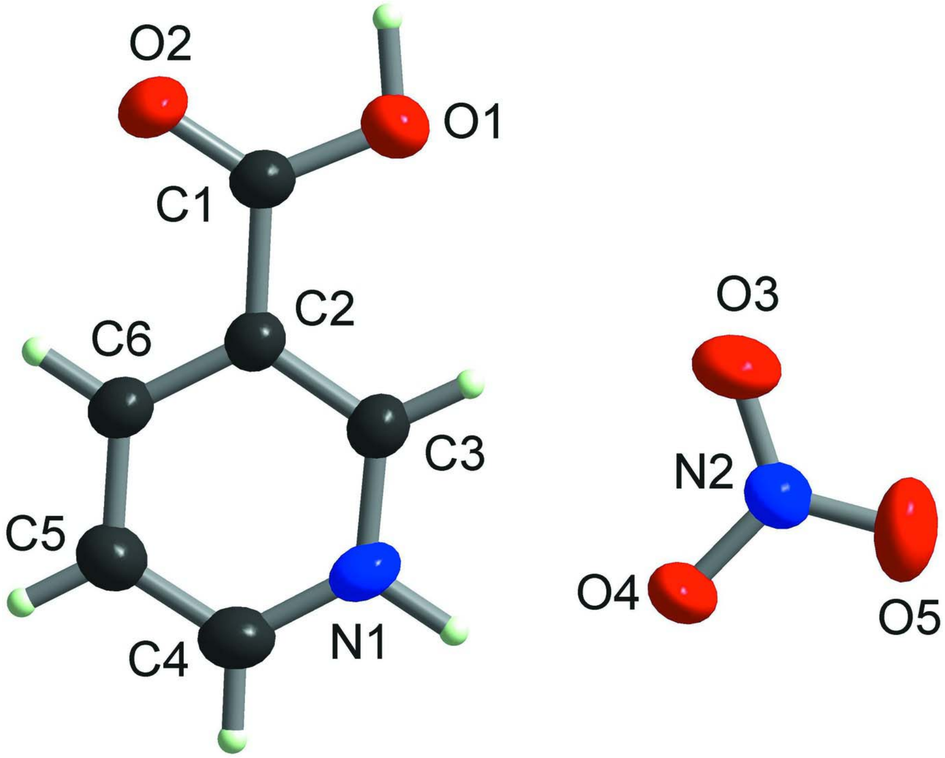

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

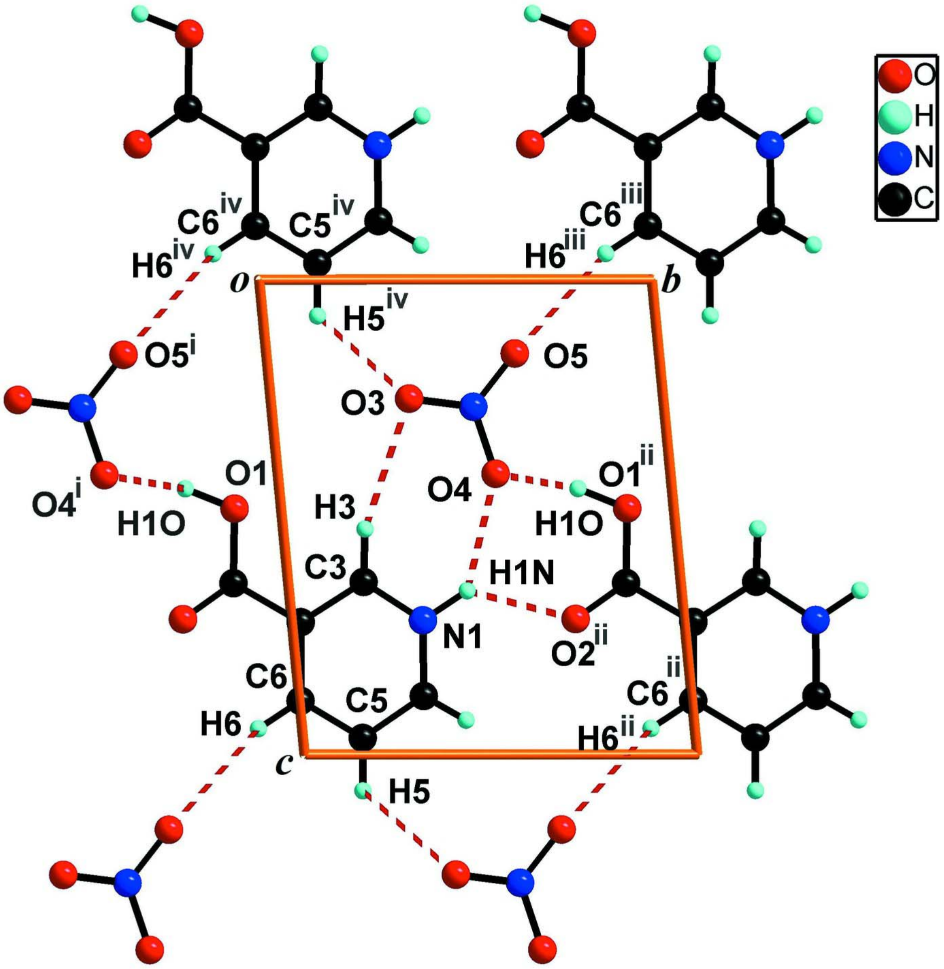

Figure 2

A view of the O—H···O, N—H···O and C—H···O interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) $x,y - 1, z$; (ii) $x, y + 1, z$; (iii) $x,y + 1, z - 1$; (iv) $x, y, z - 1$.]

3-Carboxypyridinium nitrate

Crystal data

$C_6H_6NO_2^+\cdot NO_3^-$
$M_r = 186.13$
Triclinic, $P\bar{1}$
Hall symbol: -P 1
$a = 6.7530 (4)$ Å
$b = 7.5024 (4)$ Å
$c = 8.4439 (5)$ Å
$\alpha = 81.895 (2)°$
$\beta = 82.215 (1)°$
$\gamma = 66.769 (2)°$
$V = 387.69 (4)$ Å^3

$Z = 2$
$F(000) = 192$
$D_x = 1.594$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 963 reflections
$\theta = 3.3–27.5°$
$\mu = 0.14$ mm^{-1}
$T = 293$ K
Block, colourless
0.40 × 0.20 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer	15468 measured reflections
Radiation source: fine-focus sealed tube	1760 independent reflections
Graphite monochromator	1102 reflections with $I > 2\sigma(I)$
Detector resolution: 10.0 pixels mm^{-1}	$R_{\rm int} = 0.046$
ω scans	$\theta_{\rm max} = 27.5°$, $\theta_{\rm min} = 3.3°$
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)	$h = -8\rightarrow8$
$T_{\rm min} = 0.946$, $T_{\rm max} = 0.986$	$k = -9\rightarrow9$
	$l = -10\rightarrow10$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.050$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.145$	$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2 + 0.025P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.13$	$(\Delta/\sigma)_{\rm max} < 0.001$
1760 reflections	$\Delta\rho_{\rm max} = 0.19$ e Å^{-3}
127 parameters	$\Delta\rho_{\rm min} = -0.35$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 > 2sigma(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*	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.2671 (2)	−0.1191 (2)	0.48174 (17)	0.0528 (4)
H1O	0.277 (4)	−0.232 (4)	0.443 (3)	0.079 (8)*
N1	0.2399 (3)	0.3328 (2)	0.7169 (2)	0.0461 (5)
H1N	0.245 (4)	0.449 (4)	0.654 (3)	0.086 (9)*
C1	0.2321 (3)	−0.1404 (3)	0.6385 (2)	0.0405 (5)
O2	0.2000 (3)	−0.2778 (2)	0.71433 (17)	0.0570 (5)
N2	0.2859 (3)	0.5132 (2)	0.2675 (2)	0.0461 (4)
C2	0.2350 (3)	0.0215 (2)	0.7207 (2)	0.0366 (4)
O3	0.2861 (3)	0.3523 (2)	0.2502 (2)	0.0723 (6)
C3	0.2385 (3)	0.1913 (3)	0.6360 (2)	0.0416 (5)
H3	0.2399	0.2079	0.5247	0.050*
O4	0.2781 (3)	0.5540 (2)	0.40863 (18)	0.0698 (5)
C4	0.2393 (3)	0.3167 (3)	0.8764 (3)	0.0483 (5)
H4	0.2410	0.4182	0.9273	0.058*
C5	0.2361 (3)	0.1507 (3)	0.9645 (2)	0.0497 (5)
H5	0.2348	0.1381	1.0758	0.060*

O5	0.2917 (3)	0.6324 (3)	0.1555 (2)	0.0817 (6)
C6	0.2350 (3)	0.0018 (3)	0.8862 (2)	0.0439 (5)
H6	0.2341	−0.1126	0.9449	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0802 (11)	0.0451 (9)	0.0395 (9)	−0.0309 (8)	0.0008 (7)	−0.0094 (6)
N1	0.0598 (11)	0.0313 (9)	0.0518 (11)	−0.0232 (8)	−0.0029 (8)	−0.0032 (7)
C1	0.0501 (11)	0.0346 (10)	0.0389 (11)	−0.0185 (9)	−0.0027 (8)	−0.0052 (8)
O2	0.0937 (12)	0.0416 (8)	0.0482 (9)	−0.0412 (8)	−0.0003 (8)	−0.0045 (7)
N2	0.0578 (11)	0.0445 (10)	0.0380 (9)	−0.0205 (8)	−0.0065 (7)	−0.0053 (7)
C2	0.0447 (10)	0.0304 (9)	0.0364 (10)	−0.0171 (8)	−0.0010 (8)	−0.0033 (7)
O3	0.1120 (14)	0.0623 (11)	0.0602 (11)	−0.0464 (10)	−0.0100 (9)	−0.0208 (8)
C3	0.0533 (12)	0.0346 (10)	0.0390 (10)	−0.0202 (9)	−0.0018 (8)	−0.0025 (8)
O4	0.1353 (15)	0.0522 (10)	0.0359 (9)	−0.0481 (10)	−0.0118 (9)	−0.0077 (7)
C4	0.0580 (13)	0.0421 (11)	0.0514 (13)	−0.0237 (10)	−0.0038 (9)	−0.0126 (9)
C5	0.0660 (14)	0.0477 (12)	0.0408 (12)	−0.0264 (11)	−0.0052 (10)	−0.0077 (9)
O5	0.1135 (15)	0.0740 (12)	0.0513 (10)	−0.0380 (11)	−0.0075 (9)	0.0208 (9)
C6	0.0585 (12)	0.0352 (10)	0.0419 (11)	−0.0230 (9)	−0.0024 (9)	−0.0031 (8)

Geometric parameters (Å, º)

O1—C1	1.311 (2)	N2—O4	1.261 (2)
O1—H1O	0.92 (3)	C2—C3	1.377 (2)
N1—C4	1.335 (3)	C2—C6	1.385 (3)
N1—C3	1.344 (3)	C3—H3	0.9300
N1—H1N	0.96 (3)	C4—C5	1.364 (3)
C1—O2	1.213 (2)	C4—H4	0.9300
C1—C2	1.489 (3)	C5—C6	1.379 (3)
N2—O5	1.214 (2)	C5—H5	0.9300
N2—O3	1.236 (2)	C6—H6	0.9300
C1—O1—H1O	107.7 (16)	N1—C3—C2	118.93 (18)
C4—N1—C3	123.13 (17)	N1—C3—H3	120.5
C4—N1—H1N	120.2 (16)	C2—C3—H3	120.5
C3—N1—H1N	116.7 (16)	N1—C4—C5	119.72 (18)
O2—C1—O1	124.77 (18)	N1—C4—H4	120.1
O2—C1—C2	121.11 (18)	C5—C4—H4	120.1
O1—C1—C2	114.11 (16)	C4—C5—C6	119.0 (2)
O5—N2—O3	123.04 (18)	C4—C5—H5	120.5
O5—N2—O4	119.22 (18)	C6—C5—H5	120.5
O3—N2—O4	117.74 (17)	C5—C6—C2	120.39 (18)
C3—C2—C6	118.80 (17)	C5—C6—H6	119.8
C3—C2—C1	121.64 (17)	C2—C6—H6	119.8
C6—C2—C1	119.56 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···O4^{i}	0.92 (3)	1.67 (3)	2.5833 (19)	169 (2)
N1—H1*N*···O2^{ii}	0.96 (3)	2.08 (3)	2.824 (2)	133 (2)
N1—H1*N*···O4	0.96 (3)	2.12 (3)	2.921 (2)	139 (2)
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Symmetry codes: (i) x, $y-1$, z; (ii) x, $y+1$, z; (iii) x, $y-1$, $z+1$; (iv) x, y, $z+1$.