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4-Cyanopyridinium nitrate

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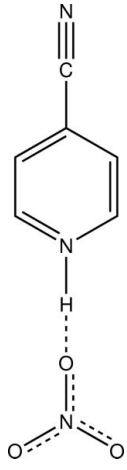
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.050; wR factor = 0.146; data-to-parameter ratio = 15.5.

The title compound, $C_6H_5N_2^+\cdot NO_3^-$, is a proton-transfer compound between 4-cyanopyridine and nitric acid. In the asymmetric unit, the components are linked by a strong N—H···O hydrogen bond. In the crystal, molecules are linked into a $C(6)$ chain along [010] by C—H···O interactions.

Related literature

For the structures and ferroelectric properties of related compounds, see: Fu *et al.* (2011*a*,*b*,*c*); Dai & Chen (2011); Xu *et al.* (2011); Zheng (2011). For graph-set motif see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_6H_5N_2^+\cdot NO_3^-$ M_r = 167.13
Monoclinic, $P2_1/c$
a = 6.3663 (2) Å
b = 13.2868 (9) Å
c = 9.1019 (2) Å
β = 103.755 (1)°
V = 747.83 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 K
0.10 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.910, T_{max} = 1.000
5151 measured reflections
1694 independent reflections
1349 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.146
S = 1.14
1694 reflections
109 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.90	1.75	2.6481 (18)	176
C4—H4A···O3^{i}	0.93	2.29	3.220 (2)	179

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

Data collection: *CrystalClear* (Rigaku, 2005); 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: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

This work was supported by a start-up grant from Southeast University, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2407).

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supporting information

Acta Cryst. (2012). E**68**, o1695 [doi:10.1107/S1600536812020697]

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S1. Comment

Simple organic salts containing strong intermolecular H-bonds have attracted an attention as materials which display ferroelectric-paraelectric phase transitions (Fu *et al.*, 2011*a*, 2011*b*, 2011*c*). With the purpose of obtaining phase transition crystals of organic salts, various organic molecules have been studied and a series of new materials have been prepared (Dai & Chen 2011; Xu, *et al.* 2011; Zheng 2011). We report here the crystal structure of the title compound. The title compound $(C_6H_5N_2)^+.NO_3^-$ is a proton-transfer compound between 4-cyanopyridine and nitric acid. In the asymmetric unit the components are linked by one strong N—H···O hydrogen bond interaction, Fig 1. In the crystal the molecules are linked into C(6) chain by simple C—H···O interactions along [010] (Bernstein, *et al.*, 1995), (Fig. 2, Table1).

S2. Experimental

The HNO_3 (5 mL), isonicotinonitrile (20 mmol) and ethanol (50 mL) were added into a 100mL flask. The mixture was stirred at 60ºC for 2 h, and then the precipitate was filtrated out. Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of the solution.

S3. Refinement

All the H atoms attached to C atoms were situated into the idealized positions and treated as riding with C–H = 0.93 Å (aromatic) with U_{iso}(H)=1.2U_{eq}(C). The positional parameters of the H atom (N) was refined freely and in the last stage of the refinement, it was restrained with the N—H = 0.90Å, with U_{iso}(H)=1.2U_{eq}(N).

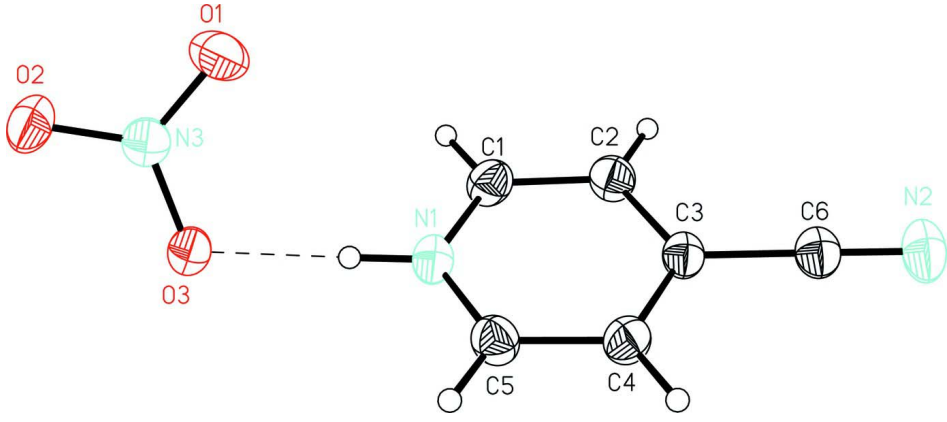

Figure 1

A view of the asymmetric unit with the atomic numbering scheme. The displacement ellipsoids were drawn at the 30% probability level.

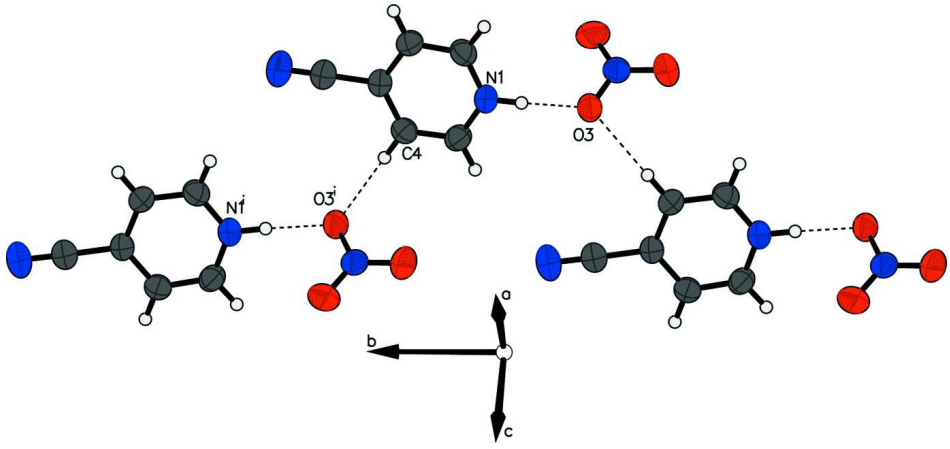

Figure 2

The crystal packing of the title compound viewed along the *a* axis showing the N—H···O and C—H···O interactions (dotted line) in the title compound. : symmetry code (i): -*x*+1, *y*+1/2, -*z*+1/2.

4-Cyanopyridinium nitrate

Crystal data

$C_6H_5N_2^+·NO_3^-$
M_r = 167.13
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 6.3663 (2) Å
b = 13.2868 (9) Å
c = 9.1019 (2) Å
β = 103.755 (1)°
V = 747.83 (6) Å^3
Z = 4
$F(000)$ = 344
D_x = 1.484 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1694 reflections
θ = 2.8–27.5°
μ = 0.12 mm^{-1}
T = 298 K
Block, colourless
0.10 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD profile fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.910, T_{max} = 1.000
5151 measured reflections
1694 independent reflections
1349 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.5°, θ_{min} = 2.8°
h = −8→7
k = −17→15
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.146
S = 1.14
1694 reflections
109 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0719P)^2 + 0.0927P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.7551 (2)	0.46541 (8)	0.20815 (14)	0.0477 (4)
N3	0.7428 (2)	0.42233 (11)	0.08092 (16)	0.0447 (4)
N1	0.7453 (2)	0.66359 (10)	0.17652 (16)	0.0440 (4)
H1	0.7413	0.5962	0.1860	0.053*
C3	0.7627 (3)	0.86509 (12)	0.13720 (18)	0.0400 (4)
N2	0.7637 (3)	1.05763 (13)	0.0946 (2)	0.0648 (5)
C4	0.6008 (3)	0.82380 (12)	0.1963 (2)	0.0456 (4)
H4A	0.4974	0.8648	0.2230	0.055*
O2	0.7350 (2)	0.32899 (10)	0.07536 (16)	0.0631 (4)
C2	0.9185 (3)	0.80337 (13)	0.0992 (2)	0.0455 (4)
H2A	1.0287	0.8304	0.0603	0.055*
C5	0.5955 (3)	0.72140 (13)	0.2149 (2)	0.0466 (4)
H5A	0.4877	0.6924	0.2543	0.056*
O1	0.7392 (3)	0.47362 (11)	−0.03265 (17)	0.0684 (5)
C1	0.9049 (3)	0.70149 (13)	0.1208 (2)	0.0474 (4)
H1A	1.0073	0.6587	0.0966	0.057*
C6	0.7672 (3)	0.97304 (14)	0.1142 (2)	0.0484 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0566 (8)	0.0384 (6)	0.0478 (7)	−0.0022 (5)	0.0118 (6)	−0.0026 (5)
N3	0.0376 (8)	0.0469 (8)	0.0511 (9)	0.0017 (6)	0.0136 (6)	−0.0006 (6)
N1	0.0483 (8)	0.0339 (7)	0.0491 (8)	−0.0012 (6)	0.0102 (6)	0.0000 (6)
C3	0.0417 (9)	0.0359 (8)	0.0399 (8)	−0.0008 (6)	0.0047 (6)	0.0005 (6)
N2	0.0756 (13)	0.0408 (9)	0.0743 (12)	−0.0017 (8)	0.0107 (10)	0.0084 (7)
C4	0.0448 (9)	0.0431 (9)	0.0513 (10)	0.0059 (7)	0.0165 (8)	0.0003 (7)
O2	0.0760 (10)	0.0423 (8)	0.0727 (10)	−0.0014 (6)	0.0211 (8)	−0.0115 (6)
C2	0.0419 (9)	0.0464 (9)	0.0506 (10)	−0.0030 (7)	0.0157 (7)	0.0009 (7)
C5	0.0448 (10)	0.0457 (10)	0.0513 (10)	−0.0031 (8)	0.0153 (8)	0.0032 (7)
O1	0.0877 (11)	0.0705 (10)	0.0542 (8)	0.0080 (8)	0.0313 (7)	0.0124 (7)
C1	0.0466 (10)	0.0447 (9)	0.0527 (10)	0.0046 (8)	0.0155 (8)	−0.0042 (7)
C6	0.0504 (11)	0.0429 (10)	0.0495 (9)	−0.0015 (7)	0.0071 (8)	0.0018 (7)

Geometric parameters (Å, º)

O3—N3	1.2774 (18)	C3—C6	1.451 (2)
N3—O1	1.234 (2)	N2—C6	1.137 (2)
N3—O2	1.2418 (19)	C4—C5	1.373 (2)
N1—C5	1.334 (2)	C4—H4A	0.9300
N1—C1	1.337 (2)	C2—C1	1.374 (2)
N1—H1	0.9005	C2—H2A	0.9300
C3—C4	1.385 (2)	C5—H5A	0.9300
C3—C2	1.392 (2)	C1—H1A	0.9300
O1—N3—O2	121.67 (16)	C3—C4—H4A	120.6
O1—N3—O3	119.80 (15)	C1—C2—C3	118.17 (15)
O2—N3—O3	118.53 (14)	C1—C2—H2A	120.9
C5—N1—C1	122.52 (15)	C3—C2—H2A	120.9
C5—N1—H1	120.6	N1—C5—C4	119.88 (16)
C1—N1—H1	116.9	N1—C5—H5A	120.1
C4—C3—C2	120.22 (15)	C4—C5—H5A	120.1
C4—C3—C6	119.33 (15)	N1—C1—C2	120.31 (16)
C2—C3—C6	120.44 (15)	N1—C1—H1A	119.8
C5—C4—C3	118.89 (15)	C2—C1—H1A	119.8
C5—C4—H4A	120.6	N2—C6—C3	177.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.90	1.75	2.6481 (18)	176
C4—H4*A*···O3^{i}	0.93	2.29	3.220 (2)	179

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