Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Pyridinium nitrate at 290 K

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Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.157
Data-to-parameter ratio = 12.1

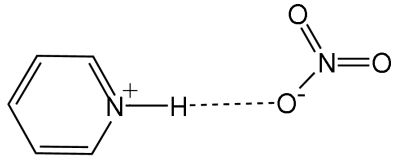
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 15 November 2004
Accepted 19 November 2004
Online 27 November 2004

A previous structural study [Serewicz *et al.* (1965). *J. Phys. Chem.* **69**, 1915–1921] of pyridinium nitrate, $C_5H_6N^+\cdot NO_3^-$, has been repeated at 290 K.

Comment

The crystal structure of pyridinium nitrate, (I), as determined by Serewicz *et al.* (1965), implied the existence of a strong hydrogen bond between the pyridinium and nitrate ions, but the precision of the data (measured at room temperature by the Weissenberg method) was insufficient to locate H atoms directly. We have redetermined this structure at two temperatures in the course of screening for materials suitable for neutron-diffraction and charge-density studies of hydrogen bonds. The 290 K structure (Fig. 1 and Table 1) is reported here. The results reported by Serewicz *et al.* (1965) are essentially confirmed, though the unit cell is slightly larger than reported previously (without s.u. values): a = 3.905, b = 12.286, c = 13.470 Å, β = 90.5° and V = 646 Å^3.



(I)

For the low-temperature results and the general discussion, see Batsanov (2004).

Experimental

The crystals of (I) were grown by slow evaporation, at room temperature, of an aqueous solution of equimolar amounts of pyridine and nitric acid.

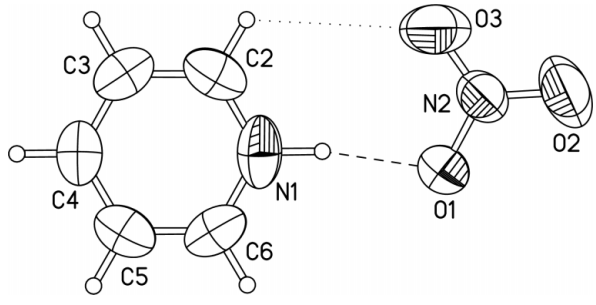


Figure 1
The molecular structure of (I) at 290 K. Displacement ellipsoids are drawn at the 50% probability level. The dashed and dotted lines indicate strong and weak hydrogen bonds, respectively.

Crystal data

$C_5H_6N^+\cdot NO_3^-$
M_r = 142.12
Monoclinic, $P2_1/c$
a = 3.9015 (6) Å
b = 12.324 (2) Å
c = 13.503 (2) Å
β = 90.57 (1)°
V = 649.2 (2) Å^3
Z = 4
D_x = 1.454 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1107 reflections
θ = 2.2–22.4°
μ = 0.12 mm^{-1}
T = 290 (2) K
Plate, colourless
0.42 × 0.37 × 0.03 mm

Data collection

Bruker APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
5258 measured reflections
1154 independent reflections
735 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 25.0°
$h = -4 \rightarrow 4$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.157
S = 1.04
1154 reflections
95 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.086P)^2 + 0.0078P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.328 (4)	C5—C6	1.337 (4)
N1—C6	1.338 (4)	O1—N2	1.251 (2)
C2—C3	1.352 (4)	O2—N2	1.217 (3)
C3—C4	1.343 (4)	O3—N2	1.225 (3)
C4—C5	1.337 (4)		
C2—N1—C6	121.0 (2)	C5—C6—N1	119.5 (2)
N1—C2—C3	119.8 (2)	O2—N2—O3	122.1 (2)
C4—C3—C2	119.1 (2)	O2—N2—O1	119.5 (2)
C5—C4—C3	120.6 (2)	O3—N2—O1	118.4 (2)
C6—C5—C4	120.0 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.94 (4)	1.86 (4)	2.787 (3)	171 (3)
N1—H1···O3	0.94 (4)	2.45 (4)	3.149 (3)	131 (3)
C2—H2···O3	0.93	2.78	3.307 (4)	117
C2—H2···O2^{i}	0.93	2.56	3.177 (3)	124
C3—H3···O2^{ii}	0.93	2.67	3.324 (3)	128
C4—H4···O3^{iii}	0.93	2.70	3.330 (3)	126
C5—H5···O3^{iii}	0.93	2.77	3.365 (4)	123
C6—H6···O1^{iv}	0.93	2.38	3.196 (3)	146
C6—H6···O2^{iv}	0.93	2.68	3.456 (3)	141

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

All H atoms were located in a difference Fourier map. Atom H1 was refined in isotropic approximation [N—H = 0.94 (4) Å], other H atoms were treated as riding in idealized positions, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The author thanks Dr I. F. Perepichka for providing single crystals of (I).

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supporting information

Acta Cryst. (2004). E**60**, o2424–o2425 [https://doi.org/10.1107/S1600536804030168]

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pyridinium nitrate

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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 3.9015 (6) Å
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c = 13.503 (2) Å
β = 90.57 (1)°
V = 649.2 (2) Å^3
Z = 4
$F(000)$ = 296
D_x = 1.454 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1107 reflections
θ = 2.2–22.4°
μ = 0.12 mm^{-1}
T = 290 K
Plate, colourless
0.42 × 0.37 × 0.03 mm

Data collection

ProteumM APEX CCD area-detector diffractometer
Radiation source: 60 W microfocus Bede Microsource with glass polycapillary optics
Graphite monochromator
Detector resolution: 8 pixels mm^{-1}
ω scans
5258 measured reflections
1154 independent reflections
735 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −4→4
k = −14→14
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.157
S = 1.04
1154 reflections
95 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.086P)^2 + 0.0078P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Special details

Experimental. The data collection nominally covered full sphere of reciprocal space, by a combination of 4 sets of ω scans, each set at different φ and/or 2θ angles and each scan (15 s exposure) covering 0.3° in ω. Crystal to detector distance 4.95 cm. Crystal decay was monitored by repeating the first 50 frames at the end of the data collection and comparing the intensities of 31 duplicate reflections.

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. H(1) atom (N-bonded) was refined in isotropic approximation (All H-atom parameters refined), other H atoms treated as riding (H-atom parameters constrained).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.3970 (5)	0.31105 (19)	0.5473 (2)	0.0746 (7)
H1	0.260 (9)	0.365 (3)	0.577 (3)	0.123 (11)*
C2	0.4746 (7)	0.2227 (3)	0.59912 (17)	0.0723 (8)
H2	0.4203	0.2188	0.6659	0.087*
C3	0.6325 (7)	0.1385 (2)	0.5545 (2)	0.0721 (8)
H3	0.6882	0.0763	0.5902	0.086*
C4	0.7081 (6)	0.1458 (2)	0.4579 (2)	0.0696 (7)
H4	0.8141	0.0879	0.4264	0.084*
C5	0.6317 (7)	0.2356 (2)	0.40669 (18)	0.0739 (8)
H5	0.6891	0.2405	0.3402	0.089*
C6	0.4739 (7)	0.3184 (2)	0.4512 (2)	0.0713 (8)
H6	0.4175	0.3805	0.4156	0.086*
O1	0.0061 (6)	0.48422 (14)	0.61596 (12)	0.0860 (7)
O2	−0.1119 (6)	0.5580 (2)	0.75437 (14)	0.1064 (8)
O3	0.1348 (6)	0.4033 (2)	0.75035 (14)	0.1090 (8)
N2	0.0100 (6)	0.48242 (17)	0.70861 (15)	0.0665 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0566 (13)	0.0572 (13)	0.110 (2)	−0.0043 (10)	0.0108 (12)	−0.0313 (14)
C2	0.0796 (18)	0.089 (2)	0.0482 (14)	−0.0210 (15)	0.0028 (12)	−0.0044 (13)
C3	0.0750 (18)	0.0580 (16)	0.0828 (19)	0.0013 (13)	−0.0170 (14)	0.0139 (13)
C4	0.0602 (16)	0.0644 (17)	0.0842 (18)	0.0078 (12)	0.0030 (13)	−0.0192 (13)
C5	0.0761 (18)	0.095 (2)	0.0507 (14)	−0.0087 (15)	0.0043 (12)	−0.0001 (13)
C6	0.0641 (16)	0.0586 (16)	0.091 (2)	−0.0029 (12)	−0.0099 (14)	0.0235 (13)
O1	0.1372 (18)	0.0713 (12)	0.0496 (10)	0.0160 (11)	0.0042 (9)	−0.0017 (7)
O2	0.129 (2)	0.1131 (16)	0.0774 (13)	0.0207 (14)	0.0102 (12)	−0.0367 (11)
O3	0.130 (2)	0.1229 (18)	0.0742 (13)	0.0350 (14)	0.0169 (12)	0.0369 (12)
N2	0.0795 (15)	0.0677 (14)	0.0524 (12)	−0.0064 (11)	0.0099 (10)	−0.0057 (10)

Geometric parameters (Å, º)

N1—C2	1.328 (4)	C4—H4	0.9300
N1—C6	1.338 (4)	C5—C6	1.337 (4)
N1—H1	0.94 (4)	C5—H5	0.9300

C2—C3	1.352 (4)	C6—H6	0.9300
C2—H2	0.9300	O1—N2	1.251 (2)
C3—C4	1.343 (4)	O2—N2	1.217 (3)
C3—H3	0.9300	O3—N2	1.225 (3)
C4—C5	1.337 (4)		

C2—N1—C6	121.0 (2)	C3—C4—H4	119.7
C2—N1—H1	119 (2)	C6—C5—C4	120.0 (2)
C6—N1—H1	120 (2)	C6—C5—H5	120.0
N1—C2—C3	119.8 (2)	C4—C5—H5	120.0
N1—C2—H2	120.1	C5—C6—N1	119.5 (2)
C3—C2—H2	120.1	C5—C6—H6	120.3
C4—C3—C2	119.1 (2)	N1—C6—H6	120.2
C4—C3—H3	120.4	O2—N2—O3	122.1 (2)
C2—C3—H3	120.5	O2—N2—O1	119.5 (2)
C5—C4—C3	120.6 (2)	O3—N2—O1	118.4 (2)
C5—C4—H4	119.7		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.94 (4)	1.86 (4)	2.787 (3)	171 (3)
N1—H1···O3	0.94 (4)	2.45 (4)	3.149 (3)	131 (3)
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C2—H2···O2^{i}	0.93	2.56	3.177 (3)	124
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C5—H5···O3^{iii}	0.93	2.77	3.365 (4)	123
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C6—H6···O2^{iv}	0.93	2.68	3.456 (3)	141

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