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2,6-Xylidinium nitrate

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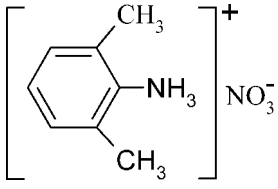
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.112; data-to-parameter ratio = 12.9.

The structure of the title organic–inorganic hybrid material, $C_8H_{12}N^+\cdot NO_3^-$, results mainly from electrostatic interactions and bifurcated N—H···O and C—H···O hydrogen bonds. Organic 2,6-xylidinium cations and inorganic nitrate anions interact to form a three-dimensional hydrogen-bond network.

Related literature

For general background, see: Xiao *et al.* (2005); Yuan *et al.* (2006); Wang & Zhang (2006). For related structures, see: Hemissi *et al.* (2001); Perpétuo & Janczak (2004). For related literature, see: Spek (2003).



Experimental

Crystal data

$C_8H_{12}N^+\cdot NO_3^-$
M_r = 184.20
Monoclinic, $P2_1/c$
a = 7.891 (2) Å
b = 8.328 (3) Å
c = 13.628 (2) Å
β = 91.47 (2)°
V = 895.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
0.20 × 0.19 × 0.17 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Absorption correction: none
3131 measured reflections
1568 independent reflections
1181 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
2 standard reflections
frequency: 120 min
intensity decay: 5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.112
S = 1.06
1568 reflections
122 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.89	2.28	3.064 (2)	147
N2—H2A···O2^{i}	0.89	2.40	3.007 (2)	126
N2—H2B···O2^{ii}	0.89	2.01	2.888 (2)	169
N2—H2C···O1	0.89	2.08	2.964 (2)	174
N2—H2C···O3	0.89	2.53	3.192 (3)	132
C5—H5···O3^{iii}	0.93	2.59	3.270 (3)	131
C8—H8B···O1	0.96	2.49	3.345 (3)	149

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2181).

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supplementary materials

Acta Cryst. (2007). E**63**, o3117 [**doi:10.1107/S1600536807025408**]

2,6-Xylidinium nitrate

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Comment

Hybrid compounds are widely investigated due to their special relevance in fundamental sciences and in several applied fields such as biomolecular sciences, catalysis and nonlinear optics (Xiao *et al.*, 2005, Yuan *et al.*, 2006). These materials are generally rich in hydrogen bonds which are considered as the most effective tool for constructing sophisticated assemblies from discrete ionic or molecular building blocks due to its strength and directionality (Wang & Zhang, 2006). In this paper, we report the synthesis and structure of a new organic nitrate. Single-crystal X-ray diffraction study of the title compounds shows that the asymmetric unit corresponds to the formula unit (I) which is made of one NO_3^- anion and one 2,6-xylidinium cation (Fig. 1). As well as electrostatic and van der Walls forces, two types of hydrogen bonds (Table 1) participate to define the crystal packing. The first one, N–H···O bonds links ammonium groups and nitrate anions into infinite layers propagating in the (b, c) plane (Fig. 2, Table 1). The second H-bonds type, C–H···O bonds, identified by *PLATON* (Spek, 2003), connects the successive layers to form a three-dimensional network (Fig. 3, Table 1). It is noteworthy that two hydrogen atoms of the NH_3 groups form bifurcated hydrogen bonds with the nitrate oxygen atoms. Bond lengths and angles observed in this structure agree well with those reported for nitrate or xylidinium compounds (Hemissi *et al.*, 2001, Perpétuo & Janczak, 2004).

Experimental

An ethanolic 2,6-xylidinium solution (5 mmol, in 5 ml) was added to an aqueous HNO_3 solution (0.5 *M*, 10 ml). The obtained solution is evaporated during several days in ambient condition until the formation of single crystals of the title compound (I).

Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms, [N–H = 0.89, C–H =0.96 Å (CH_3) with U_{iso}(H) = 1.5Ueq and C–H =0.96 Å (Ar–H), with U_{iso}(H) = 1.5Ueq]

Figures

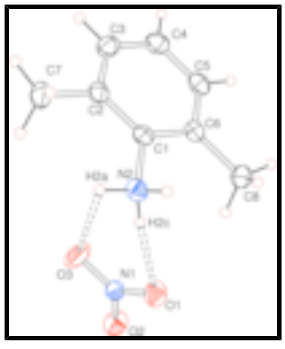

Fig. 1. *ORTEP-3* (Farrugia, 1997) view of (I) with atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level·H atoms are represented as small spheres of arbitrary radii. N—H···O hydrogen bonds are shown as dashed lines

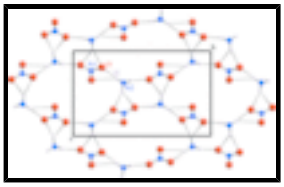

Fig. 2. Detail of a part of (011) hydrogen-bonded layer of NO_3- anion and ammonium groups in (I), with H-bonds indicated by dashed lines.

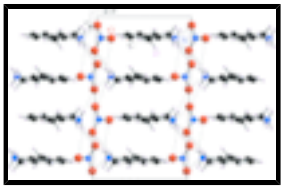

Fig. 3. Projection of (I) along *b* axis.

2,6-Xylidinium nitrate

Crystal data

$C_8H_{12}N^+·NO_3^-$	F_{000} = 392
M_r = 184.20	D_x = 1.367 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 7.891 (2) Å	θ = 8.3–9.7º
b = 8.328 (3) Å	μ = 0.11 mm^{-1}
c = 13.628 (2) Å	*T* = 293 (2) K
β = 91.47 (2)º	Parallelepiped, colourless
V = 895.2 (4) Å^3	0.20 × 0.19 × 0.17 mm
Z = 4	

Data collection

Enraf–Nonius TurboCAD-4 diffractometer	R_{int} = 0.018
Radiation source: X-ray tube	θ_{max} = 25.0º
Monochromator: graphite	θ_{min} = 2.6º
T = 293(2) K	*h* = −9→9
non–profiled ω scans	*k* = 0→9
Absorption correction: none	*l* = −16→16
3131 measured reflections	2 standard reflections
1568 independent reflections	every 120 min
1181 reflections with $I > 2\sigma(I)$	intensity decay: 5%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.037	$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.2838P]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.112$	$(\Delta/\sigma)_{max} = 0.005$
$S = 1.06$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
1568 reflections	$\Delta\rho_{min} = -0.16$ e Å^{-3}
122 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.014 (3)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.05197 (19)	0.23434 (19)	0.13088 (12)	0.0375 (4)
O1	−0.01610 (19)	0.19181 (19)	0.20863 (10)	0.0535 (4)
O2	0.0066 (2)	0.16522 (19)	0.05302 (10)	0.0548 (4)
O3	0.1585 (2)	0.3402 (2)	0.13256 (13)	0.0701 (5)
N2	0.16555 (19)	0.37350 (19)	0.36594 (11)	0.0379 (4)
C1	0.3441 (2)	0.3220 (2)	0.37321 (12)	0.0318 (4)
C2	0.4727 (2)	0.4356 (2)	0.37051 (12)	0.0366 (5)
C3	0.6386 (3)	0.3782 (3)	0.37620 (14)	0.0465 (5)
C4	0.6726 (3)	0.2168 (3)	0.38574 (15)	0.0465 (5)
C5	0.5416 (2)	0.1077 (3)	0.38996 (13)	0.0418 (5)
C6	0.3738 (2)	0.1575 (2)	0.38346 (12)	0.0341 (4)
C7	0.4382 (3)	0.6136 (3)	0.36478 (15)	0.0487 (5)
C8	0.2319 (3)	0.0377 (2)	0.38770 (16)	0.0466 (5)
H2A	0.1609	0.4793	0.3569	0.057*
H2B	0.1138	0.3482	0.4211	0.057*
H2C	0.1142	0.3241	0.3155	0.057*
H3	0.7282	0.4505	0.3735	0.056*
H4	0.7843	0.1812	0.3894	0.056*
H5	0.5660	−0.0010	0.3973	0.050*
H7A	0.3797	0.6471	0.4222	0.073*
H7B	0.3693	0.6363	0.3074	0.073*
H7C	0.5436	0.6706	0.3611	0.073*
H8A	0.2780	−0.0678	0.3983	0.070*
H8B	0.1677	0.0395	0.3269	0.070*

H8C	0.1592	0.0647	0.4407	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0347 (8)	0.0315 (9)	0.0462 (10)	0.0017 (7)	−0.0007 (7)	0.0007 (7)
O1	0.0636 (10)	0.0570 (10)	0.0401 (8)	−0.0097 (8)	0.0044 (7)	0.0044 (7)
O2	0.0665 (10)	0.0575 (10)	0.0406 (8)	−0.0133 (8)	0.0043 (7)	−0.0105 (7)
O3	0.0585 (10)	0.0629 (11)	0.0889 (13)	−0.0317 (9)	0.0005 (8)	−0.0011 (9)
N2	0.0408 (9)	0.0321 (9)	0.0406 (8)	0.0074 (7)	−0.0005 (7)	−0.0025 (7)
C1	0.0351 (9)	0.0334 (10)	0.0268 (8)	0.0049 (8)	0.0003 (7)	−0.0013 (7)
C2	0.0457 (12)	0.0355 (11)	0.0285 (9)	−0.0019 (8)	0.0010 (7)	0.0000 (8)
C3	0.0408 (11)	0.0530 (13)	0.0456 (11)	−0.0093 (10)	0.0021 (9)	0.0011 (10)
C4	0.0355 (11)	0.0572 (14)	0.0470 (12)	0.0085 (10)	0.0015 (8)	0.0018 (10)
C5	0.0473 (12)	0.0388 (11)	0.0393 (10)	0.0132 (10)	0.0006 (8)	0.0007 (9)
C6	0.0397 (10)	0.0318 (10)	0.0307 (9)	0.0043 (8)	0.0005 (7)	−0.0014 (7)
C8	0.0510 (13)	0.0319 (11)	0.0565 (12)	−0.0003 (9)	−0.0046 (9)	0.0006 (9)
C7	0.0655 (14)	0.0348 (11)	0.0457 (11)	−0.0052 (11)	0.0014 (10)	0.0026 (9)

Geometric parameters (Å, °)

O1—N1	1.251 (2)	C6—C8	1.502 (3)
O2—N1	1.251 (2)	C1—C2	1.389 (3)
N1—O3	1.218 (2)	C2—C7	1.508 (3)
N2—C1	1.473 (2)	C8—H8A	0.9600
N2—H2A	0.8900	C8—H8B	0.9600
N2—H2B	0.8900	C8—H8C	0.9600
N2—H2C	0.8900	C4—C5	1.379 (3)
C3—C4	1.376 (3)	C4—H4	0.9300
C3—C2	1.394 (3)	C5—H5	0.9300
C3—H3	0.9300	C7—H7A	0.9600
C6—C5	1.388 (3)	C7—H7B	0.9600
C6—C1	1.396 (3)	C7—H7C	0.9600
O3—N1—O1	120.00 (17)	C3—C2—C7	120.52 (19)
O3—N1—O2	122.10 (17)	C6—C8—H8A	109.5
O1—N1—O2	117.90 (16)	C6—C8—H8B	109.5
C1—N2—H2A	109.5	H8A—C8—H8B	109.5
C1—N2—H2B	109.5	C6—C8—H8C	109.5
H2A—N2—H2B	109.5	H8A—C8—H8C	109.5
C1—N2—H2C	109.5	H8B—C8—H8C	109.5
H2A—N2—H2C	109.5	C3—C4—C5	120.22 (19)
H2B—N2—H2C	109.5	C3—C4—H4	119.9
C4—C3—C2	121.37 (19)	C5—C4—H4	119.9
C4—C3—H3	119.3	C4—C5—C6	121.0 (2)
C2—C3—H3	119.3	C4—C5—H5	119.5
C5—C6—C1	117.22 (18)	C6—C5—H5	119.5
C5—C6—C8	120.64 (18)	C2—C7—H7A	109.5
C1—C6—C8	122.14 (17)	C2—C7—H7B	109.5

C2—C1—C6	123.39 (17)	H7A—C7—H7B	109.5
C2—C1—N2	119.84 (17)	C2—C7—H7C	109.5
C6—C1—N2	116.77 (16)	H7A—C7—H7C	109.5
C1—C2—C3	116.78 (18)	H7B—C7—H7C	109.5
C1—C2—C7	122.68 (18)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1^{i}	0.89	2.28	3.064 (2)	147
N2—H2A···O2^{i}	0.89	2.40	3.007 (2)	126
N2—H2B···O2^{ii}	0.89	2.01	2.888 (2)	169
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Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2; (ii) *x*, −*y*+1/2, *z*+1/2; (iii) −*x*+1, *y*−1/2, −*z*+1/2.

Fig. 1

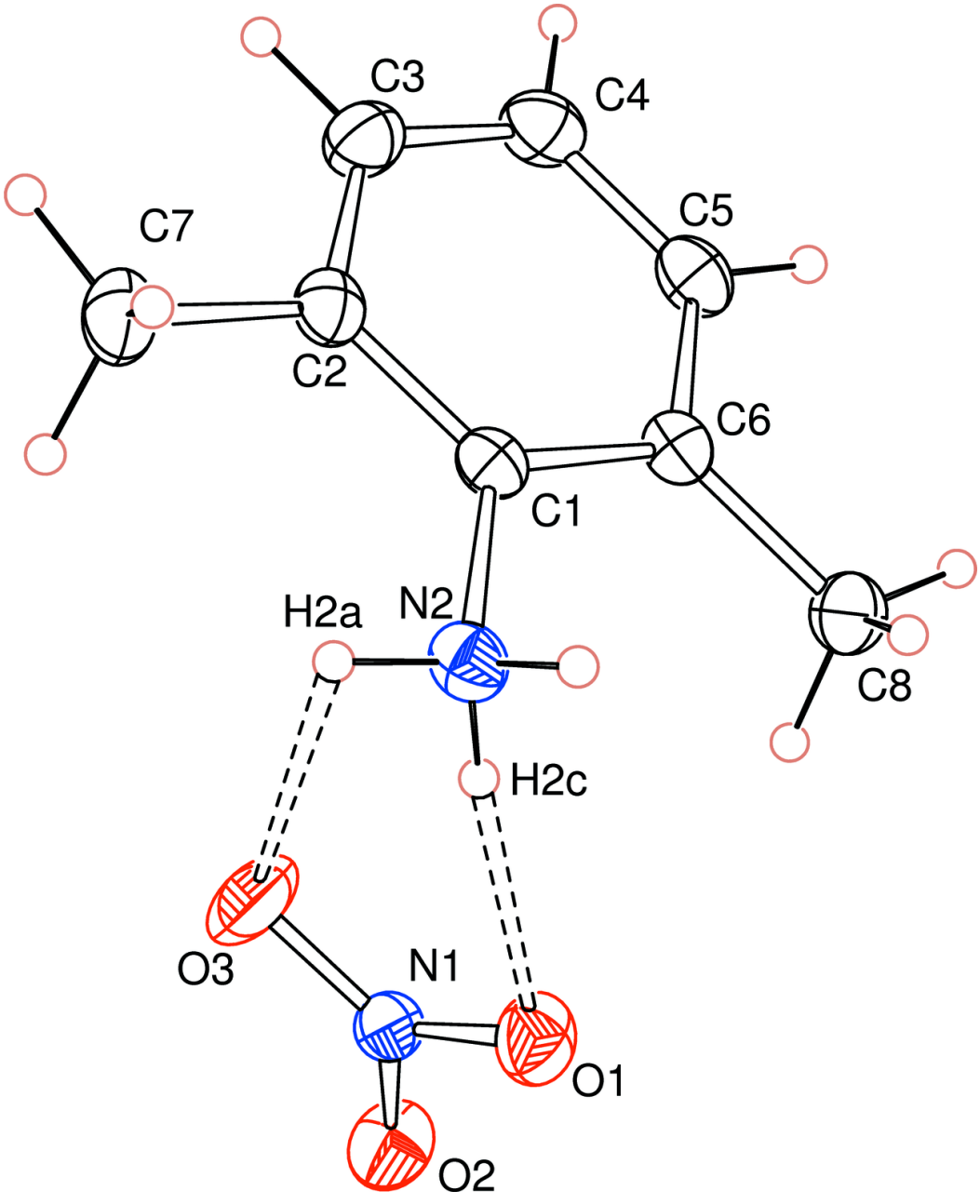

Fig. 2

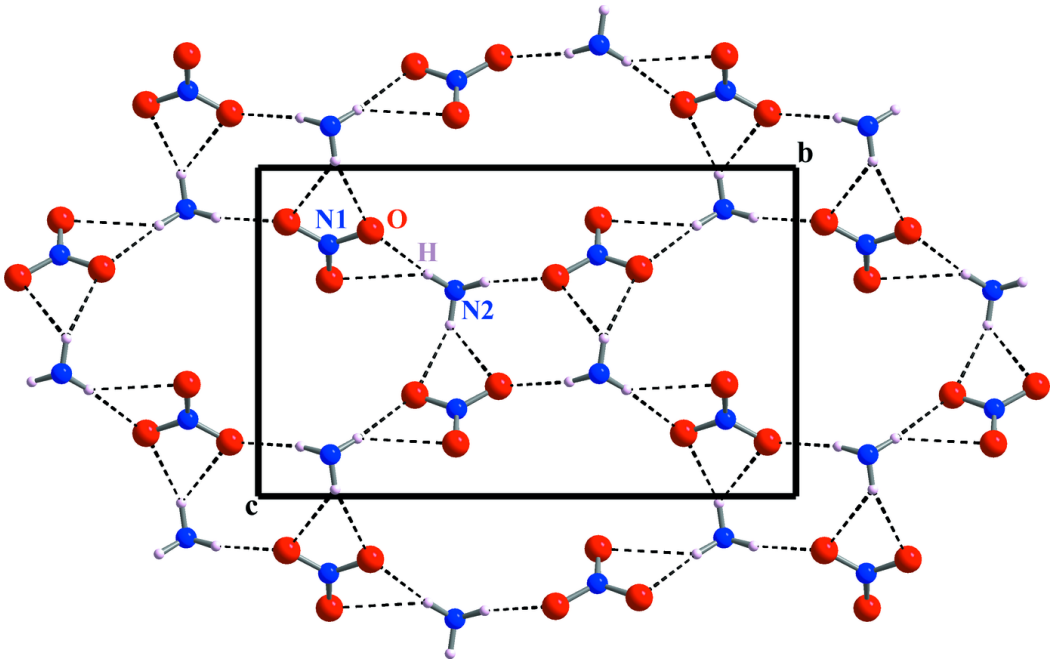

Fig. 3