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3-Aminopyridinium 3,5-dinitrobenzoate monohydrate

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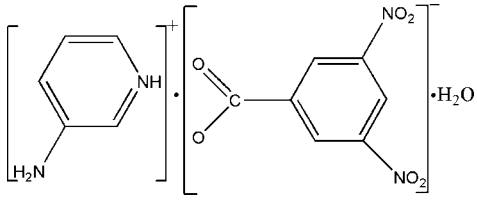
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.116; data-to-parameter ratio = 12.5.

In the title compound, $C_5H_7N_2^+\cdot C_7H_3N_2O_6^-\cdot H_2O$, the components are connected by N—H···O and O—H···O hydrogen bonds into an infinite two-dimensional network running parallel to the $(10\bar{2})$ plane.

Related literature

For a related structure, see: Wang & Wei (2007).



Experimental

Crystal data

$C_5H_7N_2^+\cdot C_7H_3N_2O_6^-\cdot H_2O$
M_r = 324.26
Monoclinic, $P2_1/c$
a = 8.904 (2) Å
b = 7.0683 (16) Å
c = 22.331 (5) Å
β = 90.002 (3)°
V = 1405.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 298 (2) K
0.40 × 0.11 × 0.09 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.950, T_{max} = 0.989
7329 measured reflections
2743 independent reflections
2110 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.116
S = 1.04
2743 reflections
220 parameters
16 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7A···O2	0.825 (10)	1.990 (10)	2.805 (2)	170 (2)
O7—H7B···O1^{i}	0.827 (9)	2.075 (10)	2.883 (2)	165 (2)
N3—H3···O1	0.883 (10)	1.730 (10)	2.6116 (19)	176 (2)
N4—H4A···O7^{ii}	0.86	2.13	2.904 (2)	149
N4—H4B···O4^{iii}	0.86	2.45	3.223 (2)	150
N4—H4B···O3^{ii}	0.86	2.59	3.110 (2)	120

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; 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: *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2550).

References

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Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wang, Z.-L. & Wei, L.-H. (2007). *Acta Cryst.* E**63**, o995–o996.

  doi:10.1107/S1600536807046740

supplementary materials

Acta Cryst. (2007). E**63**, o4174 [**doi:10.1107/S1600536807046740**]

3-Aminopyridinium 3,5-dinitrobenzoate monohydrate

L.-H. Wei

Comment

This work continues our previous synthetic and structural studies of supramolecular salts (Wang & Wei, 2007). Herein we present the crystal structure of the title salt, (I).

The title salt contains one 3-aminopyridinium cation, one 3,5-dinitrobenzoate anion and one crystallization water molecule. These components are connected by N—H···O and O—H···O hydrogen bonds, in which the water oxygen atom, two nitrogen atoms of 3-aminopyridinium cation act as the hydrogen-bond acceptor and 3-aminopyridinium cation acts as the hydrogen-bond donor (Table 1). This results in an infinite two-dimensional network runing parallel to the plane (10$\bar{2}$) (Fig. 2).

Experimental

A 5-ml ethanol soloution of 3-aminopyridine (1.0 mmol, 0.094 g) was added to an aqueous solution (25 ml) of 3,5-dinitrobenzoic acid (1.0 mmol, 0.210 g). The mixture was stirred for 10 minutes at 373 K. The solution was filtered, and the filtrate was kept at room temperature. After a week, yellow crystals of (I) were obtained.

Refinement

The crystal symmetry is monoclinic. No plausible structural models could be developed assuming orthorhombic symmetry.

The pyridine N-bound and water H atoms were located in a difference map and were refined with N—H = 0.86 (1) Å, O—H = 0.82 (1)Å and H···H = 1.39 (1) Å. The remaining H atoms were positioned geometrically with C—H = 0.93Å and N—H = 0.86 Å, and were refined as riding with U_{iso}(H) = 1.2U_{eq}(C or N) and U_{iso}(H) = 1.5U_{eq}(0).

Figures

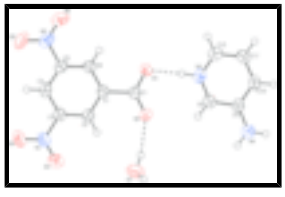

Fig. 1. The molecular structure of (I). Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

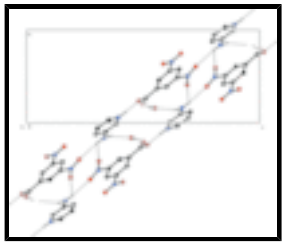

Fig. 2. The crystal packing of (I) shoiwng part of a (10$\bar{2}$) sheet. Hydrogen bonds are shown as dashed lines. For clarity, H atoms not involved in hydrogen bonds are omitted.

3-Aminopyridinium 3,5-dinitrobenzoate monohydrate

Crystal data

$C_5H_7N_2^+ \cdot C_7H_3N_2O_6^- \cdot H_2O$	$F_{000} = 672$
$M_r = 324.26$	$D_x = 1.533$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2386 reflections
$a = 8.904$ (2) Å	$\theta = 2.3$–26º
$b = 7.0683$ (16) Å	$\mu = 0.13$ mm^{-1}
$c = 22.331$ (5) Å	$T = 298$ (2) K
$\beta = 90.002$ (3)º	Block, yellow
$V = 1405.3$ (5) Å^3	0.40 × 0.11 × 0.09 mm
$Z = 4$	

Data collection

Bruker SMART APEX CCD diffractometer	2743 independent reflections
Radiation source: fine-focus sealed tube	2110 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
$T = 298$(2) K	$\theta_{max} = 26.0$º
ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$h = -10 \rightarrow 10$
$T_{min} = 0.950$, $T_{max} = 0.989$	$k = -8 \rightarrow 8$
7329 measured reflections	$l = -14 \rightarrow 27$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.116$	$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 0.2499P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
2743 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
220 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
16 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
C1	0.37418 (18)	0.5123 (2)	0.56099 (7)	0.0332 (4)
C2	0.39107 (18)	0.3634 (2)	0.60046 (8)	0.0355 (4)
H2	0.3353	0.2532	0.5957	0.043*
C3	0.49144 (19)	0.3802 (2)	0.64695 (8)	0.0366 (4)
C4	0.57995 (19)	0.5379 (2)	0.65486 (8)	0.0389 (4)
H4	0.6487	0.5459	0.6861	0.047*
C5	0.56233 (19)	0.6822 (2)	0.61467 (8)	0.0372 (4)
C6	0.46031 (19)	0.6747 (2)	0.56834 (8)	0.0369 (4)
H6	0.4492	0.7767	0.5424	0.044*
C7	0.26189 (19)	0.4982 (2)	0.51025 (7)	0.0349 (4)
N1	0.50348 (18)	0.2256 (2)	0.69057 (7)	0.0469 (4)
N2	0.65549 (18)	0.8523 (2)	0.62170 (8)	0.0508 (4)
O1	0.23633 (16)	0.64928 (17)	0.48228 (6)	0.0499 (4)
O2	0.20429 (15)	0.34363 (17)	0.49949 (6)	0.0480 (4)
O3	0.41038 (18)	0.1002 (2)	0.68925 (7)	0.0700 (5)
O4	0.60686 (18)	0.2298 (2)	0.72611 (7)	0.0693 (5)
O5	0.74764 (18)	0.8530 (2)	0.66225 (8)	0.0729 (5)
O6	0.63675 (19)	0.9837 (2)	0.58748 (8)	0.0705 (5)
O7	0.1604 (2)	−0.0161 (2)	0.54961 (7)	0.0738 (5)
H7A	0.165 (3)	0.0858 (17)	0.5317 (9)	0.081 (5)*
H7B	0.174 (3)	−0.102 (2)	0.5251 (8)	0.079 (5)*
C8	−0.00521 (19)	0.4146 (3)	0.38708 (8)	0.0382 (4)
H8	0.0140	0.3229	0.4160	0.046*
C9	−0.09708 (18)	0.3720 (2)	0.33890 (8)	0.0361 (4)
C10	−0.1206 (2)	0.5150 (3)	0.29727 (8)	0.0415 (4)
H10	−0.1807	0.4919	0.2640	0.050*
C11	−0.0566 (2)	0.6896 (3)	0.30467 (9)	0.0456 (5)
H11	−0.0740	0.7846	0.2767	0.055*
C12	0.0329 (2)	0.7240 (3)	0.35310 (9)	0.0475 (5)
H12	0.0771	0.8420	0.3585	0.057*
N3	0.05566 (17)	0.5854 (2)	0.39239 (7)	0.0417 (4)
H3	0.114 (2)	0.611 (3)	0.4234 (7)	0.060 (6)*
N4	−0.15849 (18)	0.1961 (2)	0.33348 (7)	0.0526 (5)

H4A	−0.1402	0.1110	0.3601	0.063*
H4B	−0.2155	0.1701	0.3035	0.063*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0364 (9)	0.0323 (9)	0.0310 (9)	0.0002 (7)	0.0005 (7)	−0.0019 (7)
C2	0.0363 (9)	0.0357 (9)	0.0345 (9)	−0.0023 (7)	−0.0010 (7)	−0.0011 (8)
C3	0.0399 (9)	0.0378 (9)	0.0321 (9)	0.0040 (8)	−0.0020 (7)	0.0001 (8)
C4	0.0368 (9)	0.0442 (10)	0.0358 (10)	0.0041 (8)	−0.0074 (8)	−0.0103 (8)
C5	0.0364 (9)	0.0337 (9)	0.0414 (10)	−0.0021 (7)	0.0001 (8)	−0.0091 (8)
C6	0.0416 (10)	0.0328 (9)	0.0362 (10)	0.0012 (7)	0.0020 (8)	−0.0016 (8)
C7	0.0401 (9)	0.0342 (9)	0.0303 (9)	−0.0022 (8)	−0.0027 (7)	−0.0007 (7)
N1	0.0528 (10)	0.0477 (10)	0.0401 (9)	0.0066 (8)	−0.0099 (8)	0.0034 (8)
N2	0.0517 (10)	0.0409 (10)	0.0597 (11)	−0.0054 (8)	−0.0030 (8)	−0.0135 (9)
O1	0.0694 (9)	0.0347 (7)	0.0456 (8)	−0.0054 (6)	−0.0234 (6)	0.0066 (6)
O2	0.0595 (8)	0.0364 (7)	0.0481 (8)	−0.0112 (6)	−0.0187 (6)	0.0030 (6)
O3	0.0768 (11)	0.0651 (10)	0.0682 (11)	−0.0215 (9)	−0.0165 (8)	0.0279 (8)
O4	0.0827 (11)	0.0648 (10)	0.0606 (10)	0.0067 (8)	−0.0370 (8)	0.0070 (8)
O5	0.0684 (10)	0.0626 (10)	0.0878 (13)	−0.0155 (8)	−0.0312 (9)	−0.0163 (9)
O6	0.0879 (12)	0.0435 (9)	0.0802 (12)	−0.0195 (8)	−0.0087 (9)	0.0015 (8)
O7	0.1287 (15)	0.0409 (9)	0.0519 (10)	−0.0040 (10)	−0.0016 (10)	0.0062 (7)
C8	0.0405 (10)	0.0402 (10)	0.0340 (10)	0.0025 (8)	−0.0032 (8)	0.0027 (8)
C9	0.0355 (9)	0.0399 (10)	0.0331 (9)	0.0003 (8)	−0.0011 (7)	−0.0012 (8)
C10	0.0407 (10)	0.0510 (11)	0.0327 (10)	0.0004 (8)	−0.0062 (8)	0.0009 (8)
C11	0.0499 (11)	0.0442 (11)	0.0428 (11)	−0.0006 (9)	−0.0036 (9)	0.0109 (9)
C12	0.0504 (11)	0.0415 (11)	0.0508 (12)	−0.0069 (9)	−0.0012 (9)	0.0013 (9)
N3	0.0418 (9)	0.0454 (9)	0.0379 (9)	−0.0029 (7)	−0.0077 (7)	−0.0034 (7)
N4	0.0662 (11)	0.0430 (9)	0.0487 (10)	−0.0114 (8)	−0.0167 (8)	0.0024 (8)

Geometric parameters (Å, °)

C1—C2	1.381 (2)	N2—O5	1.222 (2)
C1—C6	1.390 (2)	O7—H7A	0.825 (10)
C1—C7	1.514 (2)	O7—H7B	0.827 (9)
C2—C3	1.375 (2)	C8—N3	1.329 (2)
C2—H2	0.9300	C8—C9	1.385 (2)
C3—C4	1.376 (2)	C8—H8	0.9300
C3—N1	1.468 (2)	C9—N4	1.364 (2)
C4—C5	1.368 (3)	C9—C10	1.389 (2)
C4—H4	0.9300	C10—C11	1.370 (3)
C5—C6	1.378 (2)	C10—H10	0.9300
C5—N2	1.470 (2)	C11—C12	1.365 (3)
C6—H6	0.9300	C11—H11	0.9300
C7—O2	1.2304 (19)	C12—N3	1.330 (2)
C7—O1	1.258 (2)	C12—H12	0.9300
N1—O3	1.214 (2)	N3—H3	0.883 (10)
N1—O4	1.2157 (19)	N4—H4A	0.8600
N2—O6	1.214 (2)	N4—H4B	0.8600

C2—C1—C6	119.60 (16)	O6—N2—C5	118.76 (17)
C2—C1—C7	119.93 (15)	O5—N2—C5	117.47 (18)
C6—C1—C7	120.47 (15)	H7A—O7—H7B	108.3 (15)
C3—C2—C1	119.12 (16)	N3—C8—C9	120.51 (17)
C3—C2—H2	120.4	N3—C8—H8	119.7
C1—C2—H2	120.4	C9—C8—H8	119.7
C2—C3—C4	122.62 (17)	N4—C9—C8	120.26 (16)
C2—C3—N1	118.96 (16)	N4—C9—C10	122.92 (16)
C4—C3—N1	118.41 (16)	C8—C9—C10	116.81 (16)
C5—C4—C3	117.00 (16)	C11—C10—C9	120.80 (17)
C5—C4—H4	121.5	C11—C10—H10	119.6
C3—C4—H4	121.5	C9—C10—H10	119.6
C4—C5—C6	122.68 (16)	C12—C11—C10	119.96 (18)
C4—C5—N2	118.40 (16)	C12—C11—H11	120.0
C6—C5—N2	118.91 (17)	C10—C11—H11	120.0
C5—C6—C1	118.93 (16)	N3—C12—C11	118.68 (18)
C5—C6—H6	120.5	N3—C12—H12	120.7
C1—C6—H6	120.5	C11—C12—H12	120.7
O2—C7—O1	125.55 (16)	C8—N3—C12	123.24 (17)
O2—C7—C1	118.68 (15)	C8—N3—H3	119.5 (14)
O1—C7—C1	115.77 (15)	C12—N3—H3	117.3 (14)
O3—N1—O4	123.39 (17)	C9—N4—H4A	120.0
O3—N1—C3	118.54 (15)	C9—N4—H4B	120.0
O4—N1—C3	118.07 (17)	H4A—N4—H4B	120.0
O6—N2—O5	123.77 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7A···O2	0.825 (10)	1.990 (10)	2.805 (2)	170 (2)
O7—H7B···O1^{i}	0.827 (9)	2.075 (10)	2.883 (2)	165 (2)
N3—H3···O1	0.883 (10)	1.730 (10)	2.6116 (19)	176 (2)
N4—H4A···O7^{ii}	0.86	2.13	2.904 (2)	149
N4—H4B···O4^{iii}	0.86	2.45	3.223 (2)	150
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Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*, −*y*, −*z*+1; (iii) *x*−1, −*y*+1/2, *z*−1/2.

Fig. 1

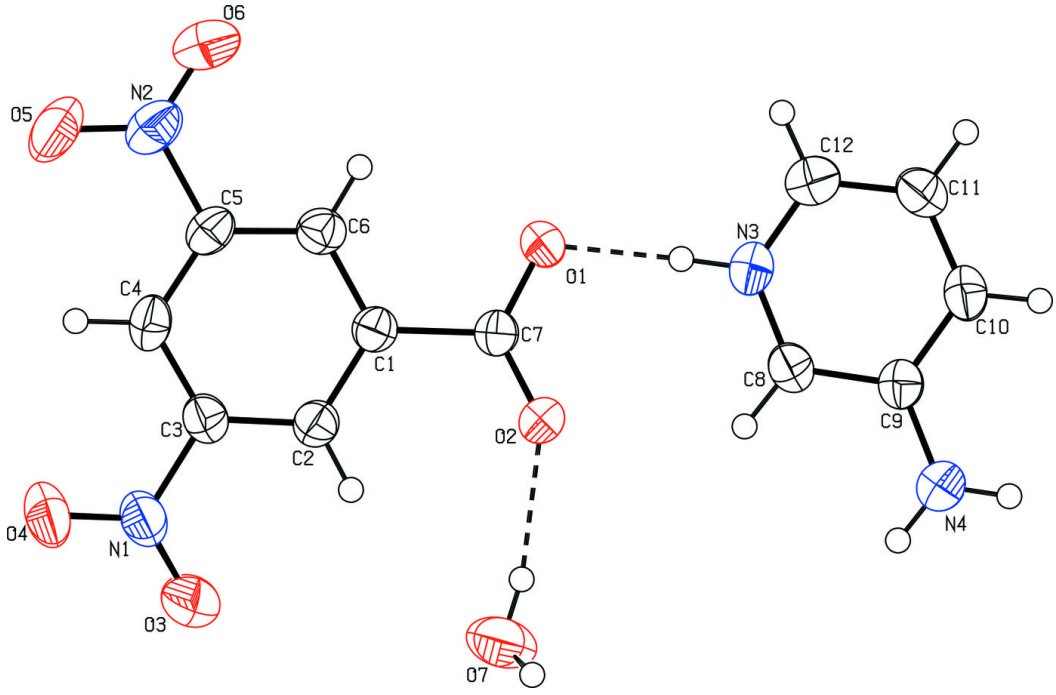

Fig. 2