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4-Acetamidoanilinium nitrate monohydrate

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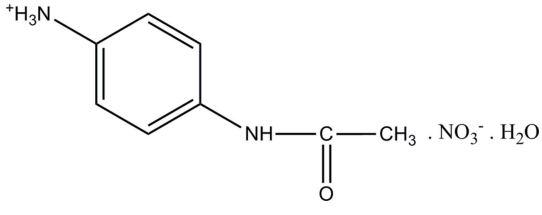
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.142; data-to-parameter ratio = 13.2.

In the title hydrated salt, $C_8H_{11}N_2O^+\cdot NO_3^-\cdot H_2O$, the N—C bond distances [1.349 (2) and 1.413 (2) Å] along with the sum of the angles (359.88°) around the acetamide N atom clearly indicate that the heteroatom has an sp^2 character. The ammonium group is involved in a total of three N—H···O hydrogen bonds, two of these are with a water molecule, which forms two O—H···O hydrogen bonds. All these hydrogen bonds link the ionic units and the water molecule into infinite planar layers parallel to (100). The remaining two N—H···O interactions in which the ammoniun group is involved link these layers into an infinite three-dimensional network.

Related literature

For the structural diversity of amine salts, see: Tooke *et al.* (2004). For related nitrate compounds, see: Dai & Chen (2011); Pourayoubi *et al.* (2011); Berrah *et al.* (2011). For hydrogen-bond patterns in related compounds, see: Flores-Alamo *et al.* (2010). For details of graph-set theory, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_{11}N_2O^+\cdot NO_3^-\cdot H_2O$
M_r = 231.21
Monoclinic, $P2_1/c$
a = 4.1059 (1) Å
b = 23.1112 (5) Å
c = 11.4702 (3) Å
β = 92.942 (1)°
V = 1087.00 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 295 K
0.41 × 0.32 × 0.25 mm

Data collection

Nonius KappaCCD diffractometer
4741 measured reflections
2606 independent reflections
1885 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.142
S = 1.06
2606 reflections
197 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.24 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
O1W—H1W···O1	0.93 (3)	1.79 (3)	2.689 (2)	161 (3)
O1W—H2W···O3	0.78 (3)	2.03 (3)	2.805 (2)	172 (3)
N1—H3···O2^{i}	0.91 (2)	2.24 (2)	3.117 (2)	161 (2)
N2—H4···O1W^{ii}	0.93 (2)	1.87 (2)	2.796 (2)	173 (2)
N2—H5···O2^{iii}	0.89 (3)	2.14 (3)	3.008 (2)	165 (3)
N2—H6···O1W^{iv}	0.93 (3)	1.95 (2)	2.827 (2)	156 (2)

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

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2060).

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4-Acetamidoanilinium nitrate monohydrate

Sana Riahi, Mohamed Lahbib Mrad, Valeria Ferretti, Frederic Lefebvre and Cherif Ben Nasr

S1. Comment

Salts of amine show interesting structural diversity governed mainly by hydrogen bonds (Tooke *et al.*, 2004), whose crystal pattern seems to be strongly influenced by the nature of the anion (Flores-Alamo *et al.*, 2010). We report here the crystal structure of one such compound, 4-acetamidoanilinium nitrate monohydrate, (I), formed from the reaction of 4-acetamidoaniline and nitric acid. The asymmetric unit of the title salt, shown in Fig. 1, contains one protonated 4-acetamidoanilinium cation, one nitrate anion and one water molecule. The NO_3^- anion geometry agrees with that observed in similar compounds (Dai & Chen, 2011; Pourayoubi *et al.*, 2011; Berrah *et al.*, 2011). In the organic cation the bond length distance N1—C7 = 1.349 (2) Å and the sum of the angles around N1 atoms = 359.88 ° clearly indicate that the heteroatom N1 has a sp^2 character. The N2 nitrogen atom is involved in a positive charge-assisted N—H···O hydrogen bond with a neighboring water molecule (N2···O1w = 2.796 (2) Å). Moreover, the water molecule forms two O—H···O interactions (O1W···O1 = 2.689 (2) Å and O1W···O3 = 2.805 (2) Å) with, respectively, the O1 oxygen atom belonging to the carbonyl group and the O3 atom of the adjacent NO_3^- anion. The weak hydrogen bond N1—H3···O2 (N1···O2 = 3.117 (2) Å) contributes to the robustness of the crystal architecture but it is not influencing the overall crystal packing pattern (Table 1, Fig. 2). Within the structure, the graph-set motif (Bernstein *et al.*, 1995) $R_4^4(22)$ is formed by two cations and two water molecules, which in turn link cations and anions to form infinite planar layers parallel to (1 0 0) (Fig. 2). These layers are interconnected via further hydrogen bonds to form a three dimensional network.

S2. Experimental

Commercial 4-acetamidobenzenamine (3 mmol) was dissolved in water/HNO_3 (50:1 *v*/*v*) solution. Colorless single crystals of the title compound, suitable for X-ray analysis, were obtained after slowly evaporation of the solvent at room temperature.

S3. Refinement

All H atoms were located in successive difference Fourier maps and refined riding on its parents atoms with U*iso*=1.2 and 1.5U*eq*(parent atom).

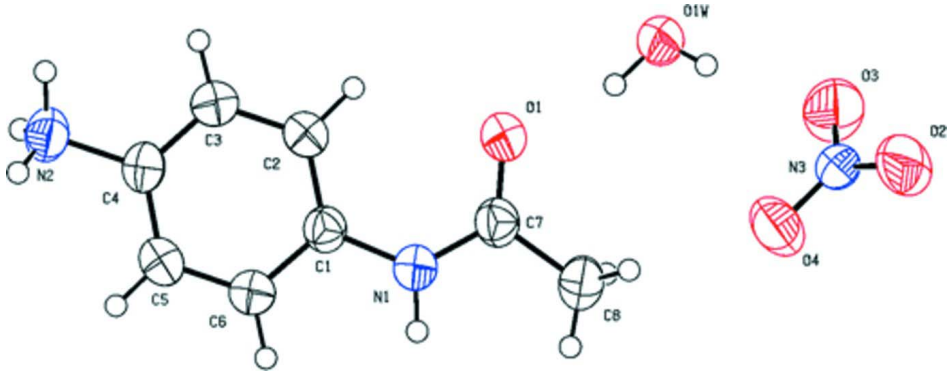

Figure 1

A view of (I) showing 50% probability displacement ellipsoids.

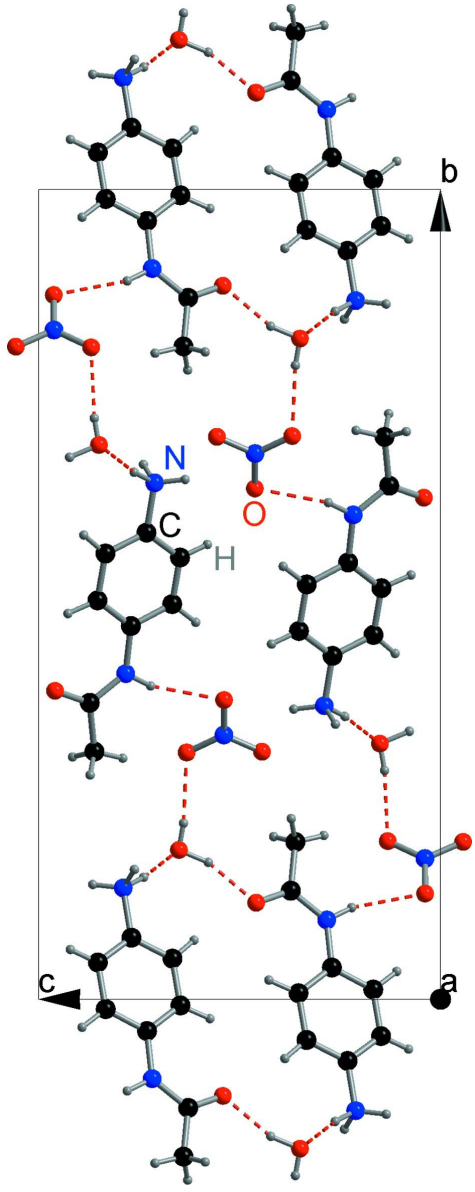

Figure 2

A view, down the *a* axis, showing a layer of the structure. The hydrogen bonding transforming the layers in a three-dimenional network has been omitted for clarity.

4-Acetamidoanilinium nitrate monohydrate

Crystal data

$C_8H_{11}N_2O^+·NO_3^-·H_2O$
M_r = 231.21
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 4.1059 (1) Å
b = 23.1112 (5) Å
c = 11.4702 (3) Å
β = 92.942 (1)°
V = 1087.00 (5) Å^3
Z = 4
F(000) = 488
D_x = 1.413 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4741 reflections
θ = 3.0–28.0°
μ = 0.12 mm^{-1}

T = 295 K
Prismatic, pale yellow

0.41 × 0.32 × 0.25 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ scans and ω scans
4741 measured reflections
2606 independent reflections

1885 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 28.0°, θ_{min} = 3.7°
h = −5→5
k = −30→27
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.142
S = 1.06
2606 reflections
197 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2 + 0.1041P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
O1	0.1624 (3)	0.11922 (5)	0.45863 (11)	0.0662 (4)
O2	0.6017 (4)	0.36949 (6)	0.53827 (14)	0.0878 (5)
O3	0.3474 (5)	0.30471 (6)	0.63123 (12)	0.0866 (5)
O4	0.3293 (4)	0.30565 (6)	0.44397 (12)	0.0766 (4)
O1W	0.2790 (3)	0.18436 (6)	0.65055 (11)	0.0570 (3)
N1	−0.0860 (3)	0.09798 (5)	0.28298 (12)	0.0500 (3)
N2	0.2062 (4)	−0.13752 (6)	0.21089 (16)	0.0543 (4)
N3	0.4244 (3)	0.32626 (5)	0.53870 (12)	0.0515 (3)
C1	0.0001 (3)	0.03922 (6)	0.27013 (12)	0.0431 (3)
C2	0.1814 (4)	0.00752 (7)	0.35317 (14)	0.0515 (4)
C3	0.2493 (4)	−0.05022 (7)	0.33325 (15)	0.0518 (4)
C4	0.1367 (3)	−0.07607 (6)	0.23129 (13)	0.0447 (3)
C5	−0.0431 (4)	−0.04540 (7)	0.14791 (15)	0.0524 (4)
C6	−0.1123 (4)	0.01213 (7)	0.16734 (14)	0.0519 (4)
C7	−0.0072 (4)	0.13406 (6)	0.37245 (13)	0.0480 (4)

C8	−0.1396 (5)	0.19416 (8)	0.3608 (2)	0.0621 (5)
H1	0.259 (4)	0.0238 (8)	0.4267 (17)	0.061 (5)*
H2	0.369 (5)	−0.0731 (9)	0.3872 (18)	0.065 (5)*
H3	−0.205 (5)	0.1131 (9)	0.2208 (18)	0.060 (5)*
H4	0.374 (5)	−0.1507 (9)	0.2619 (18)	0.067 (5)*
H5	0.243 (6)	−0.1417 (13)	0.135 (3)	0.103 (9)*
H6	0.040 (6)	−0.1603 (12)	0.239 (2)	0.095 (8)*
H7	−0.229 (4)	0.0345 (9)	0.1086 (19)	0.065 (5)*
H8	−0.123 (5)	−0.0614 (9)	0.0796 (18)	0.064 (5)*
H9	−0.251 (9)	0.2023 (17)	0.290 (4)	0.141 (12)*
H10	0.017 (8)	0.2231 (14)	0.371 (3)	0.115 (9)*
H11	−0.272 (8)	0.2008 (15)	0.415 (3)	0.124 (11)*
H1W	0.219 (6)	0.1691 (11)	0.578 (2)	0.087 (7)*
H2W	0.288 (6)	0.2175 (13)	0.639 (2)	0.088 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0952 (9)	0.0479 (7)	0.0534 (7)	0.0049 (6)	−0.0168 (6)	−0.0067 (5)
O2	0.1177 (12)	0.0606 (8)	0.0825 (10)	−0.0398 (8)	−0.0211 (8)	0.0080 (7)
O3	0.1328 (13)	0.0705 (9)	0.0578 (8)	−0.0060 (8)	0.0158 (8)	0.0140 (7)
O4	0.0939 (10)	0.0746 (9)	0.0600 (8)	−0.0196 (7)	−0.0086 (7)	−0.0111 (6)
O1W	0.0760 (8)	0.0460 (7)	0.0485 (7)	−0.0006 (5)	−0.0023 (5)	0.0004 (5)
N1	0.0605 (7)	0.0410 (7)	0.0475 (7)	0.0017 (5)	−0.0055 (6)	−0.0018 (5)
N2	0.0544 (8)	0.0424 (7)	0.0662 (10)	0.0028 (6)	0.0025 (7)	−0.0084 (6)
N3	0.0673 (8)	0.0381 (6)	0.0479 (7)	−0.0029 (5)	−0.0074 (6)	0.0047 (5)
C1	0.0473 (7)	0.0393 (7)	0.0428 (7)	−0.0037 (5)	0.0037 (6)	−0.0003 (6)
C2	0.0631 (9)	0.0466 (8)	0.0438 (8)	0.0018 (7)	−0.0059 (7)	−0.0036 (6)
C3	0.0599 (9)	0.0475 (8)	0.0472 (8)	0.0040 (7)	−0.0039 (7)	0.0018 (7)
C4	0.0451 (7)	0.0393 (7)	0.0505 (8)	−0.0025 (5)	0.0082 (6)	−0.0028 (6)
C5	0.0603 (9)	0.0493 (8)	0.0469 (9)	−0.0030 (7)	−0.0046 (7)	−0.0075 (7)
C6	0.0619 (9)	0.0460 (8)	0.0467 (8)	0.0008 (7)	−0.0074 (7)	0.0005 (7)
C7	0.0531 (8)	0.0422 (8)	0.0487 (8)	−0.0052 (6)	0.0022 (6)	−0.0018 (6)
C8	0.0674 (11)	0.0448 (9)	0.0731 (13)	0.0038 (8)	−0.0067 (10)	−0.0086 (8)

Geometric parameters (Å, º)

O1—C7	1.2286 (19)	C1—C6	1.393 (2)
O2—N3	1.2363 (18)	C2—C3	1.385 (2)
O3—N3	1.2284 (19)	C2—H1	0.96 (2)
O4—N3	1.2314 (18)	C3—C4	1.372 (2)
O1W—H1W	0.93 (3)	C3—H2	0.93 (2)
O1W—H2W	0.78 (3)	C4—C5	1.375 (2)
N1—C7	1.349 (2)	C5—C6	1.380 (2)
N1—C1	1.4129 (19)	C5—H8	0.91 (2)
N1—H3	0.91 (2)	C6—H7	0.96 (2)
N2—C4	1.4698 (19)	C7—C8	1.495 (2)
N2—H4	0.93 (2)	C8—H9	0.93 (4)

N2—H5	0.89 (3)	C8—H10	0.93 (3)
N2—H6	0.93 (3)	C8—H11	0.86 (4)
C1—C2	1.387 (2)		
H1W—O1W—H2W	103 (2)	C4—C3—H2	117.5 (13)
C7—N1—C1	128.48 (14)	C2—C3—H2	122.6 (13)
C7—N1—H3	117.0 (12)	C3—C4—C5	120.93 (14)
C1—N1—H3	114.4 (12)	C3—C4—N2	119.78 (14)
C4—N2—H4	111.1 (13)	C5—C4—N2	119.29 (14)
C4—N2—H5	107.7 (19)	C4—C5—C6	119.47 (15)
H4—N2—H5	115 (2)	C4—C5—H8	123.0 (13)
C4—N2—H6	109.9 (16)	C6—C5—H8	117.5 (13)
H4—N2—H6	97.5 (19)	C5—C6—C1	120.52 (15)
H5—N2—H6	116 (2)	C5—C6—H7	120.4 (12)
O3—N3—O4	121.44 (15)	C1—C6—H7	119.0 (12)
O3—N3—O2	120.58 (15)	O1—C7—N1	122.92 (14)
O4—N3—O2	117.98 (14)	O1—C7—C8	121.34 (15)
C2—C1—C6	119.12 (14)	N1—C7—C8	115.75 (15)
C2—C1—N1	124.34 (14)	C7—C8—H9	115 (2)
C6—C1—N1	116.53 (13)	C7—C8—H10	114.1 (18)
C3—C2—C1	120.07 (15)	H9—C8—H10	106 (3)
C3—C2—H1	117.5 (11)	C7—C8—H11	110 (2)
C1—C2—H1	122.4 (11)	H9—C8—H11	107 (3)
C4—C3—C2	119.89 (15)	H10—C8—H11	104 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*···O1	0.93 (3)	1.79 (3)	2.689 (2)	161 (3)
O1*W*—H2*W*···O3	0.78 (3)	2.03 (3)	2.805 (2)	172 (3)
N1—H3···O2^{i}	0.91 (2)	2.24 (2)	3.117 (2)	161 (2)
N2—H4···O1*W*^{ii}	0.93 (2)	1.87 (2)	2.796 (2)	173 (2)
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N2—H6···O1*W*^{iv}	0.93 (3)	1.95 (2)	2.827 (2)	156 (2)

Symmetry codes: (i) *x*−1, −*y*+1/2, *z*−1/2; (ii) −*x*+1, −*y*, −*z*+1; (iii) −*x*+1, *y*−1/2, −*z*+1/2; (iv) −*x*, −*y*, −*z*+1.