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2-(Ammoniomethyl)pyridinium sulfate monohydrate

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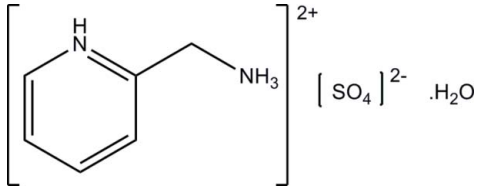
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.027; wR factor = 0.075; data-to-parameter ratio = 12.8.

In the crystal of the title hydrated molecular salt, $C_6H_{10}N_2^{2+}\cdot SO_4^{2-}\cdot H_2O$, N—H···O and O—H···O hydrogen bonds link the molecules into layers parallel to the *ab* plane. C—H···O hydrogen bonds are observed both within these layers and between molecules and ions in adjacent layers.

Related literature

For other salts of 2-aminomethylpyridine, see: Tooke *et al.* (2004); Mahjaub *et al.* (2005); Lemmerer *et al.* (2008); Khemiri *et al.* (2010); Døssing *et al.* (2001); Junk *et al.* (2006); Yuge *et al.* (2008).



Experimental

Crystal data

$C_6H_{10}N_2^{2+}\cdot SO_4^{2-}\cdot H_2O$
M_r = 224.24
Triclinic, $P\overline{1}$
a = 5.2804 (1) Å
b = 6.9458 (2) Å
c = 12.4262 (3) Å
α = 81.392 (1)°
β = 82.874 (1)°
γ = 85.193 (1)°
V = 446.15 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 100 K
0.35 × 0.34 × 0.23 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.881, T_{max} = 0.921
9334 measured reflections
1926 independent reflections
1846 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.075
S = 1.07
1926 reflections
151 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H7C···O1^{i}	0.87 (2)	2.587 (19)	2.8753 (14)	100.3 (14)
N2—H7A···O2^{ii}	0.87 (2)	1.91 (2)	2.7514 (15)	161.2 (18)
N2—H7B···O4^{iii}	0.875 (18)	1.944 (18)	2.8019 (15)	166.4 (16)
N2—H7C···O1^{iv}	0.87 (2)	1.87 (2)	2.7320 (15)	168.0 (18)
N1—H8···O4	0.86 (2)	1.86 (2)	2.7170 (15)	173.0 (18)
O5—H9···O3	0.82 (2)	1.97 (3)	2.7928 (15)	173 (2)
O5—H10···O2^{iii}	0.82 (3)	2.46 (2)	3.1822 (15)	149 (2)
O5—H10···O3^{iii}	0.82 (3)	2.55 (3)	3.3009 (16)	154 (2)
C2—H2···O3^{v}	0.93	2.39	3.2856 (16)	162
C3—H3···O5^{vi}	0.93	2.57	3.2581 (17)	131
C6—H6A···O4	0.97	2.40	3.2080 (15)	141

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT-Plus* (Bruker, 2008); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors would like to thank Alice Brink for the data collection, and the Department of Chemistry of the University of the Free State, the NRF and Sasol Ltd for funding; special thanks go to Professor Roodt and Professor Visser.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2043).

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supplementary materials

Acta Cryst. (2012). E**68**, o914 [doi:10.1107/S1600536812007714]

2-(Ammoniomethyl)pyridinium sulfate monohydrate

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Comment

2-(Ammoniomethyl)pyridinium sulfate monohydrate crystallized in the triclinic spacegroup with both nitrogen atoms protonated in the 2-aminomethylpyridine molecule. One 2-(ammoniomethyl)pyridinium cation, one sulfate anion and one solvent water molecule are present in the asymmetric unit. The bond distances compare well with those of other similar reported structures (Tooke *et al.*, 2004; Mahjaub *et al.*, 2005; Døssing *et al.*, 2001; Junk *et al.*, 2006; Yuge *et al.*, 2008; Lemmerer *et al.*, 2008; Khemiri *et al.*, 2010). Also, the four sulfur-oxygen distances of 1.4694 (17) Å, 1.4718 (12) Å, 1.4741 (13) Å and 1.4965 (13) Å are well within range for sulfur-oxygen bond distances as well as the angles of 109.58 (7) °, 110.91 (7) °, 110.63 (7) °, 109.16 (8) °, 108.32 (6) ° and 108.18 (8) °. A total of eleven hydrogen bonds (N—H···O, O—H···O and C—H···O) are observed in the crystal structure. Seven of the hydrogen bonds are between the cations and sulfate anions, three are between a water molecule and sulfate anions and one is between the water molecule and the 2-(ammoniomethyl)pyridinium cation. The sulfate anions seems to surround the 2-(ammoniomethyl)pyridinium cation. The molecules pack in alternating layers parallel with the *ab* plane (Figure 2).

Experimental

The crystal structure of (2-ammoniomethyl)pyridinium sulfate monohydrate was obtained by dissolving the ligand, 2-aminomethylpyridine, in water acidified with sulfuric acid. The final pH of the solution was recorded as pH = 6.1. The crystals were grown from this mixture by slow evaporation.

Refinement

Aromatic H atoms were positioned geometrically and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2 U_{eq}(parent) of the parent atom with a C—H distance of 0.93 Å. The methene H atoms were placed in geometrically idealized positions and constrained to ride on the parent atom with U_{iso}(H) = 1.2 U_{eq}(C) and at a distance of 0.97 Å. The O– and N–bound H atoms were placed from the electron density map and were refined freely with isotropic displacement parameters.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT-Plus* (Bruker, 2008); data reduction: *SAINT-Plus* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

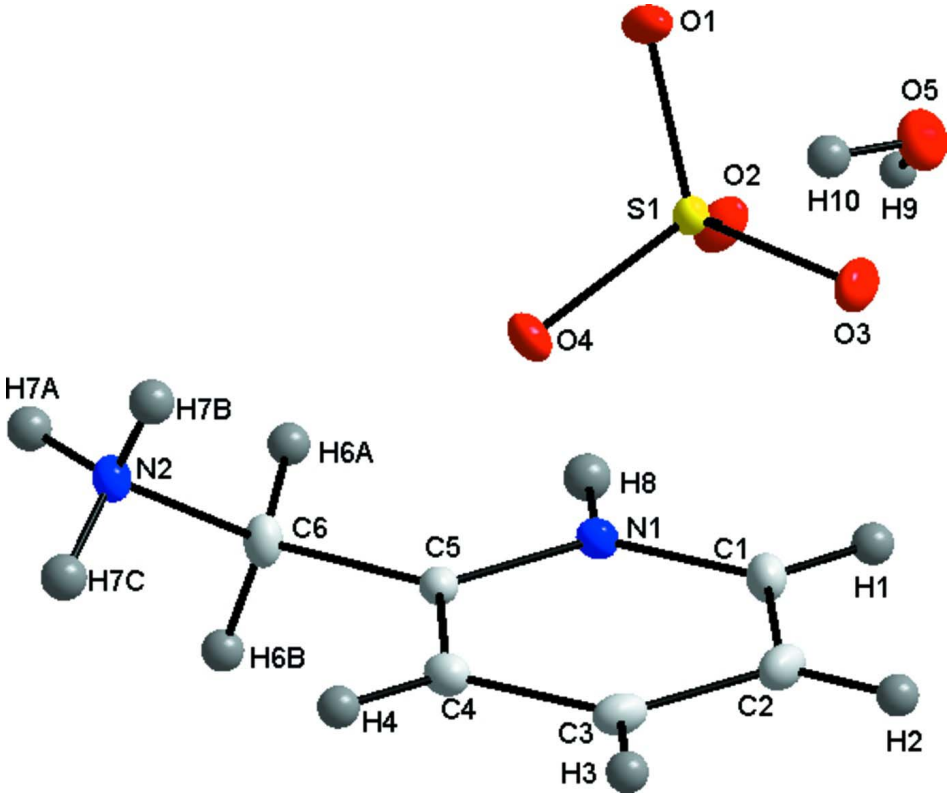

Figure 1

Diamond representation of the title compound, showing the numbering scheme and displacement ellipsoids (50% probability).

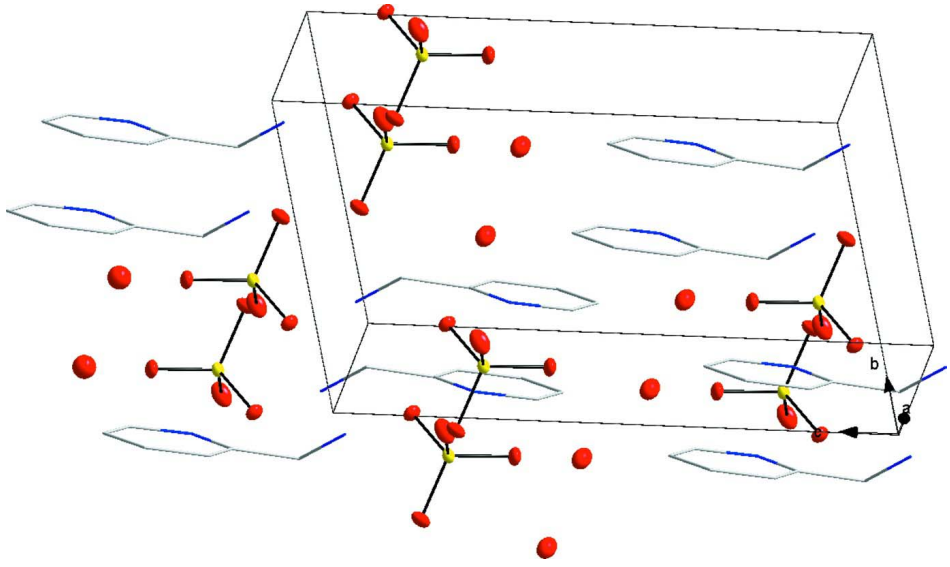

Figure 2

Packing of the title compound in the unit cell. Hydrogen atoms have been omitted for clarity.

2-(Ammoniomethyl)pyridinium sulfate monohydrate

Crystal data

$C_6H_{10}N_2^{2+}\cdot SO_4^{2-}\cdot H_2O$
$M_r = 224.24$
Triclinic, $P\overline{1}$
$a = 5.2804$ (1) Å
$b = 6.9458$ (2) Å
$c = 12.4262$ (3) Å
$\alpha = 81.392$ (1)°
$\beta = 82.874$ (1)°
$\gamma = 85.193$ (1)°
$V = 446.15$ (2) Å^3
$Z = 2$
$F(000) = 236$
$D_x = 1.669$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7388 reflections
$\theta = 3.0$–$28.4°$
$\mu = 0.36$ mm^{-1}
$T = 100$ K
Cuboid, colourless
0.35 × 0.34 × 0.23 mm

Data collection

Bruker APEXII CCD diffractometer
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.881$, $T_{max} = 0.921$
9334 measured reflections
1926 independent reflections
1846 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 27.0°$, $\theta_{min} = 3.3°$
$h = -5\rightarrow6$
$k = -8\rightarrow8$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.075$
$S = 1.07$
1926 reflections
151 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.2718P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.019$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-3}

Special details

Experimental. The intensity data were collected on a Bruker X8 ApexII 4 K Kappa CCD diffractometer using an exposure time of 10 s/frame. A total of 2250 frames were collected with a frame width of 0.5° covering up to $\theta = 28.43°$ with 97.3% completeness accomplished.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
S1	1.22129 (5)	−0.18907 (4)	0.83046 (2)	0.00951 (11)

O4	1.07225 (18)	−0.01241 (13)	0.86655 (8)	0.0137 (2)
O1	1.10127 (19)	−0.36349 (14)	0.89092 (8)	0.0170 (2)
O3	1.2139 (2)	−0.18084 (14)	0.71186 (7)	0.0171 (2)
O2	1.48757 (18)	−0.18785 (16)	0.85337 (8)	0.0206 (2)
N1	0.7557 (2)	0.15757 (15)	0.71789 (9)	0.0113 (2)
C1	0.7793 (3)	0.13486 (19)	0.61148 (11)	0.0144 (3)
H1	0.9219	0.0649	0.5815	0.017*
C2	0.5927 (3)	0.21501 (19)	0.54669 (11)	0.0148 (3)
H2	0.608	0.2008	0.4729	0.018*
C5	0.5565 (2)	0.25896 (18)	0.76561 (10)	0.0106 (2)
C4	0.3629 (2)	0.34100 (18)	0.70404 (10)	0.0121 (3)
H4	0.2225	0.4108	0.7358	0.015*
C3	0.3814 (3)	0.31756 (18)	0.59413 (11)	0.0137 (3)
H3	0.2518	0.3707	0.5522	0.016*
C6	0.5689 (2)	0.27891 (19)	0.88357 (10)	0.0124 (3)
H6A	0.6844	0.1755	0.9149	0.015*
H6B	0.6376	0.4026	0.8875	0.015*
O5	0.8069 (2)	−0.35284 (16)	0.64843 (9)	0.0215 (2)
N2	0.3142 (2)	0.26919 (17)	0.94897 (9)	0.0116 (2)
H9	0.924 (5)	−0.307 (3)	0.6720 (18)	0.034 (6)*
H10	0.682 (5)	−0.305 (3)	0.6830 (19)	0.041 (6)*
H7B	0.235 (3)	0.172 (3)	0.9344 (14)	0.014 (4)*
H7C	0.226 (4)	0.380 (3)	0.9352 (15)	0.023 (4)*
H7A	0.341 (4)	0.246 (3)	1.0179 (17)	0.023 (5)*
H8	0.864 (4)	0.100 (3)	0.7607 (15)	0.021 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.00975 (17)	0.00982 (17)	0.00827 (17)	0.00146 (11)	−0.00037 (11)	−0.00074 (11)
O4	0.0147 (4)	0.0112 (4)	0.0154 (4)	0.0017 (3)	−0.0013 (3)	−0.0048 (3)
O1	0.0185 (5)	0.0109 (4)	0.0181 (5)	0.0016 (4)	0.0055 (4)	0.0017 (4)
O3	0.0247 (5)	0.0183 (5)	0.0090 (5)	−0.0038 (4)	−0.0010 (4)	−0.0035 (4)
O2	0.0097 (5)	0.0313 (6)	0.0186 (5)	0.0013 (4)	−0.0033 (4)	0.0036 (4)
N1	0.0120 (5)	0.0098 (5)	0.0115 (5)	0.0013 (4)	−0.0014 (4)	−0.0009 (4)
C1	0.0165 (6)	0.0132 (6)	0.0128 (6)	−0.0004 (5)	0.0019 (5)	−0.0033 (5)
C2	0.0202 (6)	0.0143 (6)	0.0099 (6)	−0.0041 (5)	−0.0006 (5)	−0.0010 (5)
C5	0.0120 (6)	0.0088 (5)	0.0108 (6)	−0.0019 (4)	0.0005 (4)	−0.0015 (4)
C4	0.0116 (6)	0.0105 (6)	0.0140 (6)	−0.0006 (4)	−0.0008 (5)	−0.0012 (5)
C3	0.0153 (6)	0.0119 (6)	0.0138 (6)	−0.0039 (5)	−0.0042 (5)	0.0019 (5)
C6	0.0108 (6)	0.0154 (6)	0.0112 (6)	0.0005 (4)	−0.0007 (5)	−0.0039 (5)
O5	0.0183 (5)	0.0250 (5)	0.0229 (5)	−0.0029 (4)	−0.0016 (4)	−0.0087 (4)
N2	0.0119 (5)	0.0120 (5)	0.0108 (5)	0.0008 (4)	−0.0003 (4)	−0.0029 (4)

Geometric parameters (Å, º)

S1—O2	1.4697 (10)	C5—C6	1.5028 (16)
S1—O3	1.4715 (9)	C4—C3	1.3900 (18)
S1—O1	1.4737 (10)	C4—H4	0.93
S1—O4	1.4971 (9)	C3—H3	0.93

N1—C5	1.3419 (16)	C6—N2	1.4835 (16)
N1—C1	1.3443 (17)	C6—H6A	0.97
N1—H8	0.86 (2)	C6—H6B	0.97
C1—C2	1.3783 (19)	O5—H9	0.82 (2)
C1—H1	0.93	O5—H10	0.82 (3)
C2—C3	1.3891 (19)	N2—H7B	0.875 (18)
C2—H2	0.93	N2—H7C	0.87 (2)
C5—C4	1.3860 (17)	N2—H7A	0.87 (2)
O2—S1—O3	109.59 (6)	C5—C4—H4	120.5
O2—S1—O1	110.90 (6)	C3—C4—H4	120.5
O3—S1—O1	110.65 (6)	C2—C3—C4	120.26 (12)
O2—S1—O4	109.13 (6)	C2—C3—H3	119.9
O3—S1—O4	108.33 (6)	C4—C3—H3	119.9
O1—S1—O4	108.17 (5)	N2—C6—C5	112.19 (10)
C5—N1—C1	122.78 (11)	N2—C6—H6A	109.2
C5—N1—H8	116.0 (12)	C5—C6—H6A	109.2
C1—N1—H8	121.1 (12)	N2—C6—H6B	109.2
N1—C1—C2	120.15 (12)	C5—C6—H6B	109.2
N1—C1—H1	119.9	H6A—C6—H6B	107.9
C2—C1—H1	119.9	H9—O5—H10	101 (2)
C1—C2—C3	118.50 (12)	C6—N2—H7B	110.1 (11)
C1—C2—H2	120.8	C6—N2—H7C	109.4 (13)
C3—C2—H2	120.8	H7B—N2—H7C	111.4 (17)
N1—C5—C4	119.20 (11)	C6—N2—H7A	106.9 (12)
N1—C5—C6	115.55 (11)	H7B—N2—H7A	108.3 (16)
C4—C5—C6	125.22 (11)	H7C—N2—H7A	110.7 (17)
C5—C4—C3	119.09 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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