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3-Hydroxyanilinium hydrogensulfate

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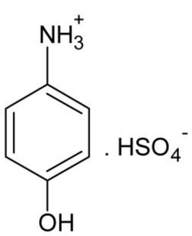
Key indicators: single-crystal X-ray study; *T* = 293 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.030; *wR* factor = 0.099; data-to-parameter ratio = 18.5.

In the title compound, $C_6H_8NO^+\cdot HSO_4^-$, there is an intricate cation–cation, cation–anion and anion–anion three-dimensional hydrogen-bond network.

Related literature

For related literature, see: Aakeroy *et al.* (1999); Benali-Cherif, Allouche *et al.* (2007); Benali-Cherif, Direm *et al.* (2007); Cherouana *et al.* (2003); Hagrman *et al.* (1999); Mazeaud *et al.* (2000); Soghomonian *et al.* (1995).



Experimental

Crystal data

$C_6H_8NO^+\cdot HSO_4^-$
M_r = 207.21
Monoclinic, $P2_1$
a = 7.3142 (3) Å
b = 5.8612 (2) Å
c = 9.8969 (2) Å
β = 98.829 (2)°
V = 419.25 (2) Å^3
Z = 2
Mo *K*α radiation
μ = 0.38 mm^{-1}
T = 293 (2) K
0.15 × 0.12 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
5851 measured reflections
2241 independent reflections
2150 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.099
S = 1.14
2241 reflections
121 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Absolute structure: Flack (1983), with 1095 Friedel pairs
Flack parameter: −0.02 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^i	0.89	2.03	2.830 (2)	149
N1—H1*B*···O2^{ii}	0.89	1.98	2.853 (2)	166
N1—H1*C*···O3^{iii}	0.89	2.14	2.962 (2)	152
N1—H1*C*···O4	0.89	2.39	2.975 (2)	124
O41—H41···O4^{iv}	0.82	1.88	2.695 (2)	174
O1—H1···O41^{iv}	0.82	1.86	2.642 (2)	160

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

Data collection: *KappaCCD Server Software* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2197).

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supplementary materials

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3-Hydroxyanilinium hydrogensulfate

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Comment

Crystal engineering of organic-inorganic hybrid compounds is currently of great interest and these matrials have received increasing attention during the past few decades (Mazeaud *et al.*, 2000; Soghomonian *et al.*, 1995) owing to their interesting structural topologies and potential application in materials science, such as ion-exchange, adsorption, molecular recognition, catalysis and magnetism (Aakeroy *et al.*, 1999; Hagrman *et al.*, 1999). The crystal structure of *m*-hydroxyanilinuim bisulfate, (I), was determined as part of our investigations on the structural characteristics of organic-inorganic layered compounds and an ongoing study on D—H···A hydrogen-bonding in systems of hybrid materials including anilinium derivatives such as 4-Carboxyanilinium hydrogensulfate (Benali-Cherif, Direm *et al.*, 2007), 2-carboxyanilinium dihydrogenphosphate (Benali-Cherif, Allouche *et al.*, 2007) and *m*-Carboxyphenylanilinium bisulfate (Cherouana, *et al.*, 2003),

The asymmetric unit of (I) contains a monoprotonated *p*-hydroxyanilinium cation and bisulfate anion (Figure 1). Intra atomic bond distance and angles in the title compound shows the monprotonation of the organic entity and confirms the presence of the bisulfate (HSO_4^-) anion.

The crystal structure of the title compound is built up from intricate cation-cation, cation-anion and anion-anion hydrogen-bonds interaction in a three-dimensional network. Strong and moderate N—H···N, N—H···O, O—H···O hydrogen bonding ensure the cohesion of the crystal through the formation of three-dimensional hydrogen bond network and the strongest one are observed between cation and anion (O41—H···O4 2.695 (2) Å, (O1—H1···O41 2.642 (2) Å)). Principal hydrogen bonding values are listed in Table 1, and the interactions are illustrated in Fig. 2.

Experimental

Single crystals of the title compound are prepared by slow evaporation at room temperature of an aqueous solution of 4-hydroxyaniline acid C_6H_7NO and sulfate acid (H_2SO_4).

Refinement

The OH and NH_3 H-atoms of the anion and cation entities were located in difference Fourier syntheses and refined as riding atoms with distances constraints of N—H = 0.89 Å and O—H = 0.82 Å [U_{iso} (H) = 1.5U_{eq}(N,*O*)]. Aromatic H atoms were located in difference Fourier syntheses and were allowed to ride on their parent C atoms with C—H = 0.93 Å and U_{iso} = 1.2U_{eq}(C).

Figures

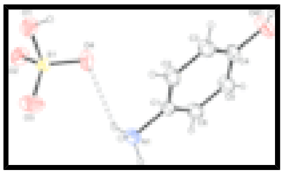

Fig. 1. View of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H bond is shown as dashed line. H atoms are represented as small spheres of arbitrary radii.

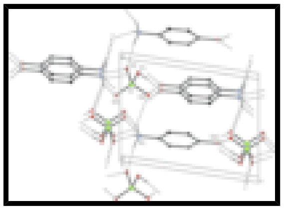

Fig. 2. Partial packing view showing the three dimensionnal hydrogen-bonding network.

3-Hydroxyanilinium hydrogensulfate

Crystal data

$C_6H_8NO^+{\cdot}HS_1O_4^-$	F_{000} = 216
M_r = 207.21	D_x = 1.641 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 5862 reflections
a = 7.3142 (3) Å	θ = 2.1–30.0°
b = 5.8612 (2) Å	μ = 0.38 mm^{-1}
c = 9.8969 (2) Å	T = 293 (2) K
β = 98.829 (2)°	Prism, colourless
V = 419.25 (2) Å^3	0.15 × 0.12 × 0.10 mm
Z = 2	

Data collection

Nonius KappaCCD area-detector diffractometer	2150 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.043
Monochromator: graphite	θ_{max} = 30.0°
T = 293(2) K	θ_{min} = 2.1°
ω/θ scans	h = −8→10
Absorption correction: none	k = −7→8
5851 measured reflections	l = −13→13
2241 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained

$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2 + 0.051P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.099$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.14$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
2241 reflections	$\Delta\rho_{min} = -0.45$ e Å^{-3}
121 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with 1095 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.02 (8)
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}
C1	0.2336 (2)	0.1606 (4)	0.28583 (15)	0.0237 (3)
C2	0.3143 (3)	0.3498 (4)	0.35270 (18)	0.0280 (4)
H2	0.3587	0.4675	0.3037	0.034*
C3	0.3287 (3)	0.3631 (3)	0.49435 (19)	0.0281 (4)
H3	0.3824	0.4902	0.5408	0.034*
C4	0.2629 (2)	0.1865 (4)	0.56539 (15)	0.0249 (3)
C5	0.1808 (3)	−0.0052 (4)	0.4976 (2)	0.0310 (4)
H5	0.1367	−0.1234	0.5463	0.037*
C6	0.1661 (3)	−0.0165 (4)	0.35594 (19)	0.0297 (4)
H6	0.1112	−0.1424	0.3088	0.036*
N1	0.2226 (2)	0.1419 (3)	0.13659 (15)	0.0271 (3)
H1A	0.2343	0.2798	0.1012	0.041*
H1B	0.1137	0.0826	0.1011	0.041*
H1C	0.3130	0.0520	0.1171	0.041*
O41	0.2772 (3)	0.1876 (3)	0.70515 (13)	0.0385 (3)
H41	0.2966	0.3181	0.7336	0.058*
O2	0.8476 (2)	0.0097 (3)	0.04947 (17)	0.0373 (4)
O1	0.8468 (2)	0.3880 (3)	0.13117 (15)	0.0333 (3)
H1	0.8085	0.4545	0.1941	0.050*
O3	0.5869 (2)	0.2501 (3)	−0.03018 (17)	0.0474 (5)
O4	0.6326 (2)	0.1115 (3)	0.20212 (15)	0.0334 (3)

S1	0.71804 (5)	0.17750 (8)	0.08525 (3)	0.02299 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0227 (6)	0.0291 (9)	0.0187 (6)	0.0027 (8)	0.0019 (5)	−0.0021 (7)
C2	0.0348 (10)	0.0259 (9)	0.0246 (8)	−0.0047 (8)	0.0085 (7)	0.0005 (7)
C3	0.0340 (9)	0.0245 (9)	0.0260 (8)	−0.0040 (7)	0.0056 (7)	−0.0035 (7)
C4	0.0294 (7)	0.0255 (8)	0.0202 (6)	0.0047 (9)	0.0046 (5)	−0.0006 (8)
C5	0.0390 (10)	0.0290 (10)	0.0253 (8)	−0.0072 (8)	0.0061 (7)	0.0017 (7)
C6	0.0341 (10)	0.0280 (10)	0.0261 (8)	−0.0056 (8)	0.0015 (7)	−0.0035 (7)
N1	0.0283 (6)	0.0325 (10)	0.0202 (6)	0.0031 (7)	0.0029 (5)	−0.0008 (6)
O41	0.0659 (9)	0.0314 (7)	0.0192 (5)	−0.0074 (9)	0.0092 (5)	−0.0022 (6)
O2	0.0310 (7)	0.0397 (9)	0.0432 (8)	0.0016 (7)	0.0120 (6)	−0.0167 (7)
O1	0.0339 (7)	0.0315 (8)	0.0366 (7)	−0.0064 (6)	0.0115 (6)	−0.0055 (6)
O3	0.0462 (9)	0.0469 (9)	0.0423 (8)	−0.0053 (8)	−0.0144 (7)	0.0167 (7)
O4	0.0407 (7)	0.0331 (8)	0.0303 (6)	−0.0025 (6)	0.0176 (6)	0.0004 (5)
S1	0.02315 (18)	0.0258 (2)	0.02035 (18)	0.00081 (18)	0.00441 (12)	0.00186 (16)

Geometric parameters (Å, °)

C1—C2	1.377 (3)	C6—H6	0.9300
C1—C6	1.382 (3)	N1—H1A	0.8900
C1—N1	1.4709 (19)	N1—H1B	0.8900
C2—C3	1.392 (3)	N1—H1C	0.8900
C2—H2	0.9300	O41—H41	0.8200
C3—C4	1.379 (3)	O2—S1	1.4467 (16)
C3—H3	0.9300	O1—S1	1.5759 (15)
C4—O41	1.3710 (18)	O1—H1	0.8200
C4—C5	1.396 (3)	O3—S1	1.4389 (15)
C5—C6	1.391 (3)	O4—S1	1.4491 (14)
C5—H5	0.9300		
C2—C1—C6	121.59 (15)	C1—C6—H6	120.3
C2—C1—N1	119.74 (18)	C5—C6—H6	120.3
C6—C1—N1	118.66 (18)	C1—N1—H1A	109.5
C1—C2—C3	119.31 (17)	C1—N1—H1B	109.5
C1—C2—H2	120.3	H1A—N1—H1B	109.5
C3—C2—H2	120.3	C1—N1—H1C	109.5
C4—C3—C2	119.56 (17)	H1A—N1—H1C	109.5
C4—C3—H3	120.2	H1B—N1—H1C	109.5
C2—C3—H3	120.2	C4—O41—H41	109.5
O41—C4—C3	122.2 (2)	S1—O1—H1	109.5
O41—C4—C5	116.7 (2)	O3—S1—O2	112.88 (11)
C3—C4—C5	121.13 (16)	O3—S1—O4	113.33 (10)
C6—C5—C4	118.94 (19)	O2—S1—O4	113.12 (10)
C6—C5—H5	120.5	O3—S1—O1	107.06 (10)
C4—C5—H5	120.5	O2—S1—O1	102.60 (9)
C1—C6—C5	119.47 (18)	O4—S1—O1	106.89 (9)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O2^{i}	0.89	2.03	2.830 (2)	149
N1—H1B···O2^{ii}	0.89	1.98	2.853 (2)	166
N1—H1C···O3^{iii}	0.89	2.14	2.962 (2)	152
N1—H1C···O4	0.89	2.39	2.975 (2)	124
O41—H41···O4^{iv}	0.82	1.88	2.695 (2)	174
O1—H1···O41^{iv}	0.82	1.86	2.642 (2)	160

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

Fig. 1

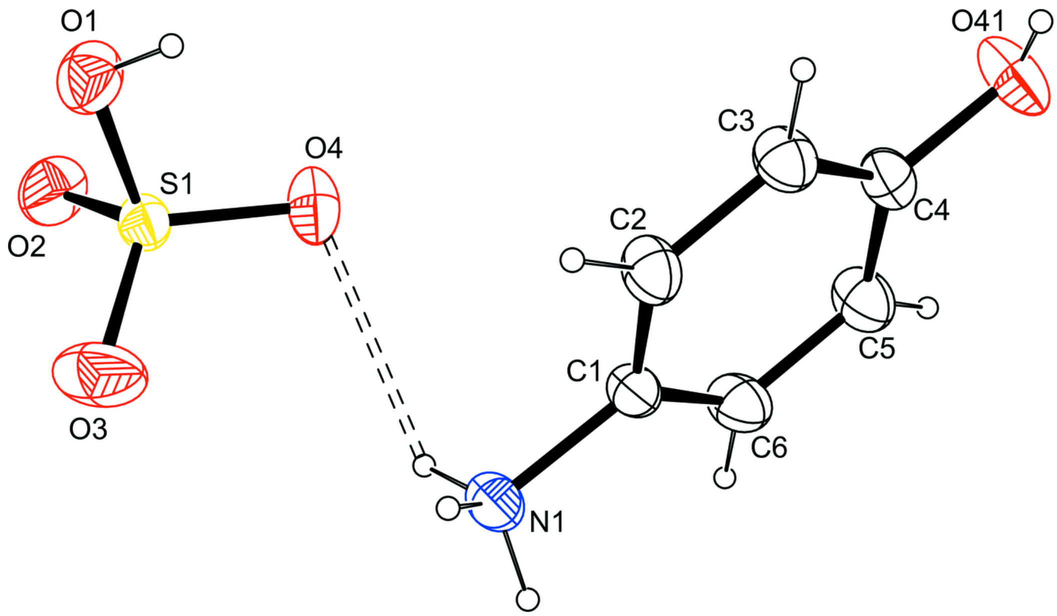

Fig. 2