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2-(Morpholinium-4-yl)ethylammonium sulfate methanol monosolvate

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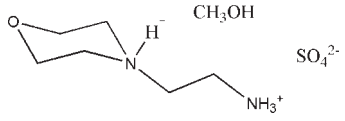
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.087; wR factor = 0.286; data-to-parameter ratio = 16.3.

In the title compound, $C_6H_{16}N_2O^{2+}\cdot SO_4^{2-}\cdot CH_3OH$, the morpholinium ring of the dication adopts a chair conformation. The crystal structure is stabilized by an extensive three-dimensional network of intermolecular O—H···O, N—H···O, O—H···S and N—H···S hydrogen bonds.

Related literature

For supramolecular compounds derived from the self-assembly of inorganic acids with organic amines, see: Xu (2010); Akhtar *et al.* (2010); Zhang & Liu (2010); Hemamalini & Fun (2010); SiMa (2010).



Experimental

Crystal data

$C_6H_{16}N_2O^{2+}\cdot SO_4^{2-}\cdot CH_4O$
M_r = 260.31
Monoclinic, $P2_1/c$
a = 15.593 (14) Å
b = 8.573 (8) Å
c = 9.483 (9) Å
β = 106.395 (11)°
V = 1216.0 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 298 K
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.946, T_{max} = 0.951
5674 measured reflections
2462 independent reflections
1726 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.087
$wR(F^2)$ = 0.286
S = 1.08
2462 reflections
151 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O4^{i}	0.82	1.85	2.659 (6)	172
O5—H5···S1^{i}	0.82	2.92	3.636 (6)	147
N2—H2A···O1^{ii}	0.89	2.07	2.914 (5)	158
N2—H2A···O3^{ii}	0.89	2.30	3.001 (5)	135
N2—H2A···S1^{ii}	0.89	2.68	3.553 (4)	167
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N2—H2C···O2	0.89	2.18	2.997 (5)	153
N2—H2C···O1	0.89	2.23	2.930 (4)	135
N2—H2C···S1	0.89	2.71	3.565 (4)	162
N1—H1···O2^{iv}	0.90 (1)	2.01 (3)	2.837 (5)	152 (5)
N1—H1···O4^{iv}	0.90 (1)	2.60 (3)	3.382 (6)	146 (5)
N1—H1···S1^{iv}	0.90 (1)	2.85 (1)	3.738 (4)	172 (5)

Symmetry codes: (i) $x, y, z+1$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2757).

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supporting information

Acta Cryst. (2010). E**66**, o951 [doi:10.1107/S1600536810010846]

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S1. Comment

Self-assembly of inorganic acids with organic amines readily gives rise to hydrogen-bonded supramolecular compounds (Xu, 2010; Akhtar *et al.*, 2010; Zhang & Liu, 2010; Hemamalini & Fun, 2010; SiMa, 2010). In order to construct a similar supramolecular compound, the title compound was prepared from the reaction of 2-morpholin-4-ylethylamine with sulfuric acid in a methanol solution and its structure is reported here.

The title compound consists of a 2-morpholin-4-ylethylammonium dication, a sulfate dianion, and a methanol molecule (Fig. 1). The crystal structure is stabilized by intermolecular O–H···O, N–H···O, O–H···S, and N–H···S hydrogen bonds (Table 1, Fig. 2).

S2. Experimental

Equimolar quantities (1.0 mmol each) of 2-morpholin-4-ylethylamine and sulfuric acid were mixed in a methanol solution. The mixture was stirred at room temperature for half an hour to give a colorless solution. After keeping the solution in air for a few days, colorless block-shaped crystals were formed.

S3. Refinement

H1 attached to N1 was located from a difference map and refined isotropically, with the N–H distance restrained to 0.90 (1) Å. Other H atoms were placed in calculated positions and constrained to ride on their parent atoms with C–H distances of 0.96-0.97 Å, N–H distances of 0.89 Å, O–H distance of 0.82 Å, and with U_{iso}(H) set to 1.2U_{eq}(C,*N*) and 1.5U_{eq}(O5 and C7). Crystals were small and very weakly diffracting and this is reflected in the low fraction of measured reflections and the relatively poor residuals.

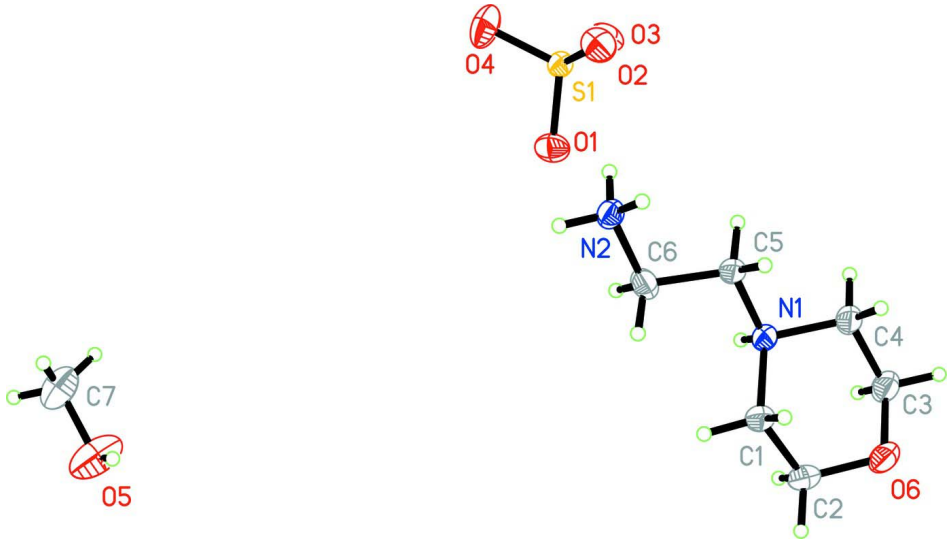

Figure 1

The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

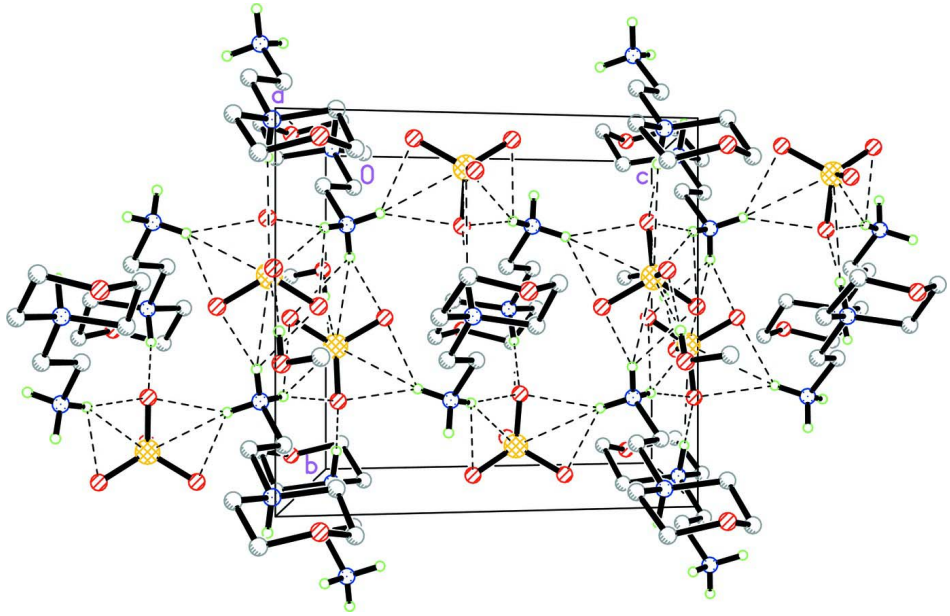

Figure 2

Molecular packing of the title compound, viewed along the a axis. Hydrogen bonds are shown as dashed lines.

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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
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c = 9.483 (9) Å
β = 106.395 (11)°
V = 1216.0 (19) Å^3
Z = 4
F(000) = 560
D_x = 1.422 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2104 reflections

θ = 2.2–27.7°
μ = 0.28 mm^{-1}
T = 298 K

Block, colorless
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.946, T_{max} = 0.951

5674 measured reflections
2462 independent reflections
1726 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 27.0°, θ_{min} = 2.7°
h = −19→14
k = −10→10
l = −10→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.087
$wR(F^2)$ = 0.286
S = 1.08
2462 reflections
151 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.1965P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.35803 (6)	0.59946 (11)	0.06772 (9)	0.0369 (4)
O1	0.3987 (2)	0.5157 (3)	0.2047 (3)	0.0523 (8)
O2	0.3969 (2)	0.7579 (3)	0.0786 (3)	0.0557 (8)
O3	0.3768 (2)	0.5166 (4)	−0.0531 (3)	0.0574 (9)
O4	0.2617 (2)	0.6167 (5)	0.0429 (5)	0.0772 (11)
O5	0.1552 (3)	0.3688 (5)	1.0088 (7)	0.1071 (18)
H5	0.1834	0.4507	1.0182	0.161*
O6	0.9107 (2)	0.4451 (4)	0.5902 (3)	0.0606 (9)
N1	0.7246 (2)	0.5104 (4)	0.4511 (3)	0.0371 (7)
N2	0.51382 (19)	0.7510 (4)	0.3894 (3)	0.0403 (8)
H2A	0.5402	0.8420	0.3841	0.061*
H2B	0.4754	0.7621	0.4426	0.061*

H2C	0.4846	0.7192	0.2993	0.061*
C1	0.7681 (3)	0.5356 (5)	0.6131 (4)	0.0469 (10)
H1A	0.7247	0.5183	0.6673	0.056*
H1B	0.7893	0.6422	0.6300	0.056*
C2	0.8462 (3)	0.4232 (7)	0.6667 (5)	0.0593 (12)
H2D	0.8736	0.4392	0.7709	0.071*
H2E	0.8242	0.3168	0.6528	0.071*
C3	0.8723 (3)	0.4061 (6)	0.4418 (5)	0.0558 (12)
H3A	0.8513	0.2991	0.4351	0.067*
H3B	0.9174	0.4135	0.3895	0.067*
C4	0.7949 (3)	0.5129 (5)	0.3698 (4)	0.0471 (10)
H4A	0.8168	0.6186	0.3682	0.056*
H4B	0.7684	0.4801	0.2690	0.056*
C5	0.6558 (2)	0.6314 (5)	0.3848 (4)	0.0394 (8)
H5A	0.6304	0.6094	0.2810	0.047*
H5B	0.6841	0.7330	0.3938	0.047*
C6	0.5817 (3)	0.6351 (5)	0.4589 (5)	0.0497 (10)
H6A	0.5542	0.5328	0.4524	0.060*
H6B	0.6065	0.6604	0.5621	0.060*
C7	0.0750 (4)	0.3876 (9)	0.9066 (8)	0.0911 (19)
H7A	0.0295	0.3351	0.9386	0.137*
H7B	0.0778	0.3444	0.8146	0.137*
H7C	0.0611	0.4968	0.8944	0.137*
H1	0.702 (4)	0.413 (3)	0.437 (6)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0318 (6)	0.0445 (6)	0.0335 (6)	0.0004 (3)	0.0079 (4)	0.0002 (3)
O1	0.0618 (19)	0.0560 (18)	0.0355 (14)	−0.0019 (14)	0.0078 (13)	0.0054 (11)
O2	0.068 (2)	0.0439 (17)	0.0592 (18)	−0.0076 (14)	0.0246 (14)	−0.0018 (12)
O3	0.071 (2)	0.065 (2)	0.0364 (14)	0.0049 (15)	0.0159 (13)	−0.0076 (12)
O4	0.0357 (18)	0.089 (3)	0.107 (3)	0.0122 (15)	0.0190 (18)	0.005 (2)
O5	0.070 (3)	0.081 (3)	0.143 (4)	−0.002 (2)	−0.016 (3)	0.016 (3)
O6	0.0330 (15)	0.092 (2)	0.0519 (17)	0.0065 (15)	0.0046 (12)	−0.0012 (16)
N1	0.0305 (15)	0.0446 (17)	0.0346 (15)	−0.0036 (12)	0.0068 (11)	−0.0012 (12)
N2	0.0317 (16)	0.0509 (19)	0.0376 (15)	−0.0020 (12)	0.0085 (12)	−0.0046 (13)
C1	0.039 (2)	0.067 (3)	0.0326 (18)	0.0013 (18)	0.0064 (14)	0.0002 (16)
C2	0.043 (2)	0.089 (3)	0.039 (2)	0.009 (2)	0.0008 (17)	0.011 (2)
C3	0.036 (2)	0.081 (3)	0.050 (2)	0.0041 (19)	0.0122 (17)	−0.0046 (19)
C4	0.036 (2)	0.068 (3)	0.0380 (18)	−0.0050 (18)	0.0119 (15)	−0.0039 (16)
C5	0.0333 (18)	0.055 (2)	0.0287 (16)	0.0000 (15)	0.0072 (13)	−0.0001 (14)
C6	0.050 (2)	0.059 (2)	0.047 (2)	0.0108 (19)	0.0257 (17)	0.0136 (18)
C7	0.049 (3)	0.108 (5)	0.109 (5)	0.005 (3)	0.011 (3)	0.000 (4)

Geometric parameters (Å, º)

S1—O3	1.446 (3)	C1—H1A	0.9700
S1—O4	1.461 (4)	C1—H1B	0.9700
S1—O1	1.463 (3)	C2—H2D	0.9700
S1—O2	1.479 (3)	C2—H2E	0.9700
O5—C7	1.358 (7)	C3—C4	1.516 (6)
O5—H5	0.8200	C3—H3A	0.9700
O6—C3	1.405 (5)	C3—H3B	0.9700
O6—C2	1.409 (6)	C4—H4A	0.9700
N1—C5	1.497 (5)	C4—H4B	0.9700
N1—C4	1.508 (5)	C5—C6	1.512 (5)
N1—C1	1.509 (5)	C5—H5A	0.9700
N1—H1	0.899 (10)	C5—H5B	0.9700
N2—C6	1.466 (5)	C6—H6A	0.9700
N2—H2A	0.8900	C6—H6B	0.9700
N2—H2B	0.8900	C7—H7A	0.9600
N2—H2C	0.8900	C7—H7B	0.9600
C1—C2	1.524 (6)	C7—H7C	0.9600
O3—S1—O4	110.5 (2)	H2D—C2—H2E	108.0
O3—S1—O1	109.1 (2)	O6—C3—C4	111.5 (4)
O4—S1—O1	111.2 (2)	O6—C3—H3A	109.3
O3—S1—O2	109.6 (2)	C4—C3—H3A	109.3
O4—S1—O2	107.5 (2)	O6—C3—H3B	109.3
O1—S1—O2	108.85 (17)	C4—C3—H3B	109.3
C7—O5—H5	109.5	H3A—C3—H3B	108.0
C3—O6—C2	108.6 (3)	N1—C4—C3	111.3 (3)
C5—N1—C4	108.3 (3)	N1—C4—H4A	109.4
C5—N1—C1	113.0 (3)	C3—C4—H4A	109.4
C4—N1—C1	109.6 (3)	N1—C4—H4B	109.4
C5—N1—H1	112 (4)	C3—C4—H4B	109.4
C4—N1—H1	104 (4)	H4A—C4—H4B	108.0
C1—N1—H1	109 (4)	N1—C5—C6	111.7 (3)
C6—N2—H2A	109.5	N1—C5—H5A	109.3
C6—N2—H2B	109.5	C6—C5—H5A	109.3
H2A—N2—H2B	109.5	N1—C5—H5B	109.3
C6—N2—H2C	109.5	C6—C5—H5B	109.3
H2A—N2—H2C	109.5	H5A—C5—H5B	107.9
H2B—N2—H2C	109.5	N2—C6—C5	110.8 (3)
N1—C1—C2	109.6 (3)	N2—C6—H6A	109.5
N1—C1—H1A	109.7	C5—C6—H6A	109.5
C2—C1—H1A	109.7	N2—C6—H6B	109.5
N1—C1—H1B	109.7	C5—C6—H6B	109.5
C2—C1—H1B	109.7	H6A—C6—H6B	108.1
H1A—C1—H1B	108.2	O5—C7—H7A	109.5
O6—C2—C1	111.3 (4)	O5—C7—H7B	109.5
O6—C2—H2D	109.4	H7A—C7—H7B	109.5

C1—C2—H2D	109.4	O5—C7—H7C	109.5
O6—C2—H2E	109.4	H7A—C7—H7C	109.5
C1—C2—H2E	109.4	H7B—C7—H7C	109.5

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