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Morpholinium hydrogensulfate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
Disorder in solvent or counterion
R factor = 0.043
wR factor = 0.103
Data-to-parameter ratio = 8.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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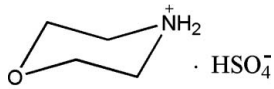
The title compound, $C_4H_{10}NO^+\cdot HSO_4^-$, contains a morpholinium cation and a hydrogensulfate anion, both placed on a mirror plane and with the anion disordered over two positions. Strong N—H···O hydrogen bonds play a dominant role in the crystal packing.

Comment

The title compound, (I), was unexpectedly obtained during an attempt to synthesize a Tb^{3+} complex (see *Experimental*) including 2-thienoyltrifluoroacetone as ligand (Yang & Wang, 1985).



(I)

Selected geometric parameters for (I) are listed in Table 1. An ellipsoid plot of the component ions is shown in Fig. 1. The asymmetric unit contains a morpholinium cation, which adopts a chair conformation, and a hydrogensulfate anion. Both ions lie on a mirror plane. The hydrogensulfate anion is disordered over two positions and anchored to the cation through a quite strong N1—H1···O13 interaction (Table 2 and Fig. 2). In addition, an N1—H1···O1 hydrogen bond links symmetry-related cations into infinite one-dimensional chains arranged along [100] (Fig. 2).

Experimental

Morpholine (1.52 ml) was added to ethanol (25 ml) containing 2-thienoyltrifluoroacetone (3.90 g, 17.6 mM). This mixture was refluxed

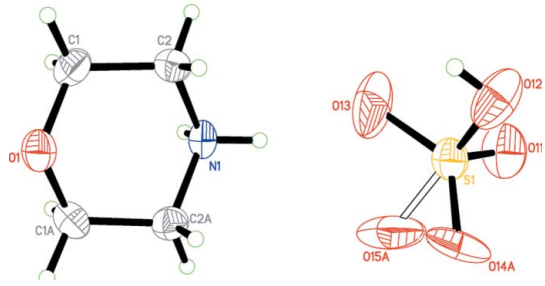


Figure 1
A view of the structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. In the anion, atoms S1 and O11 lie on the mirror plane; only one mirror-equivalent of each other O atom is shown, and the only displayed aspect of the disorder is the pair of atoms (O14A and O15A), which are alternative sites with equal occupancy factors. [Symmetry code: (A) x, y, $\frac{1}{2}-z$.]

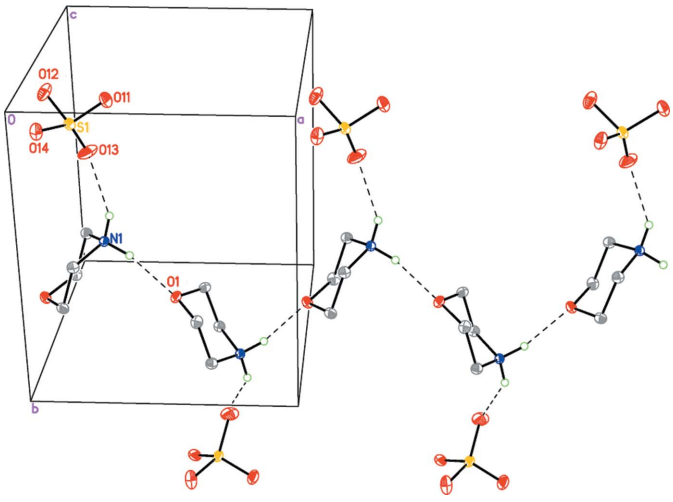


Figure 2
A packing view of (I). One disordered position of the anion has been omitted for clarity. H atoms not involved in hydrogen bonding have been omitted and hydrogen bonds are represented as dashed lines.

and $Tb_2(SO_4)_3$ dissolved in hot ethanol (25 ml) was added. The reaction system was cooled to 298 K, giving (I), which was washed with ethanol and dried. An ethanol solution of (I) was allowed to evaporate slowly at 298 K for several days, giving colourless crystals suitable for X-ray studies.

Crystal data

$C_4H_{10}NO^+\cdot HSO_4^-$
M_r = 185.20
Orthorhombic, *Pnam*
a = 8.2829 (7) Å
b = 9.5671 (9) Å
c = 9.7824 (9) Å
V = 775.19 (12) Å^3
Z = 4
D_x = 1.587 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 298 (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.858, T_{max} = 0.925
2960 measured reflections
720 independent reflections
714 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.103
S = 1.15
720 reflections
81 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 0.5414P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.073 (6)

Table 1
Selected geometric parameters (Å, °).

S1—O14	1.366 (9)	S1—O12	1.493 (7)
S1—O11	1.389 (3)	N1—C2	1.476 (3)
S1—O15	1.390 (10)	C1—O1	1.421 (3)
S1—O13	1.441 (6)	C1—C2	1.499 (4)
C2^i—N1—C2	110.4 (3)	N1—C2—C1	109.3 (2)
O1—C1—C2	110.3 (2)	C1—O1—C1^i	110.2 (3)
O1—C1—C2—N1	58.5 (3)		

Symmetry code: (i) $x, y, -z+\frac{1}{2}$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{ii}	0.90	2.01	2.887 (4)	166
N1—H1D···O13	0.90	2.07	2.823 (7)	141

Symmetry code: (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+\frac{1}{2}$.

All H atoms were placed in calculated positions and allowed to ride on their parent atoms, with U_{iso}(H) values set at 1.2U_{eq}(parent atom); constrained distances: C—H = 0.97 Å, N—H = 0.90 Å and O—H = 0.82 Å. In the anion, O14 and O15 are closely-spaced alternative sites with equal occupancies; the extensive disorder also involves mirror-equivalent atoms as alternatives. Finally, in order to obtain a sensible geometry, three bond lengths were restrained in the anion, *viz*. S1—O12 = 1.58 (1) Å, S1—O14 = 1.41 (1) Å and S1—O15 = 1.41 (1) Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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