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Bis(L-threoninium) sulfate monohydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.032
wR factor = 0.094
Data-to-parameter ratio = 8.1

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

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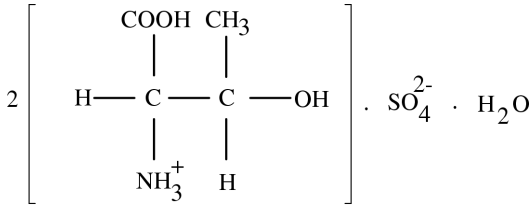
In the title compound, $2C_4H_{10}NO_3^+\cdot SO_4^{2-}\cdot H_2O$, both threoninium molecules have a *gauche* II form for the C^γ and *gauche* I form for the O^γ. The sulfate anion links the cation in an infinite manner through hydrogen bonds along the *b* and *c* axes. The two water molecules on the twofold axes link the sulfate groups and one of the cations.

Comment

Threonine is the isometric form of amino acids containing more than one asymmetric C atom. The crystal structures of DL-threonine (Shoemaker *et al.*, 1950), L-threonine (Shoemaker *et al.*, 1950) and L-allothreonine (Swaminathan & Srinivasan, 1975) have been reported. In the present study, the threonine complex with sulfuric acid, (I), has been investigated.

The geometries of the L-threoninium cations *A* and *B* are as expected (Fig. 1 and Table 1). In cation *A*, the O1*A*—C11—C12—N11 and O1*B*—C11—C12—N11 torsion angles are −12.6 (4) and 167.9 (3)°, respectively, and the corresponding torsion angles in *B* are −8.3 (4) and 172.1 (3)°. This tendency towards non-planarity is also found in various amino acids (Lakshiminarayanan *et al.*, 1967). The side-chain conformation is given by the torsion angles about $C^\alpha—C^\beta$, giving the orientation of the γ atom with respect to N (Lakshiminarayanan *et al.*, 1967). These angles are close to 60, 180 and 300°. In the present case, the C^γ atom moves to a *gauche* II form [−49.2 (4) and −48.1 (4)°] and the O^γ atom to a *gauche* I form [75.8 (3) and 78.6 (3)°] for both molecules.



(I)

The sulfate anion forms hydrogen bonds with threoninium molecules *A* and *B* (Fig. 2 and Table 2). Threonium molecule *A* is engaged in a three-centred zigzag (Z1) head-to-tail sequence with N11—H11*C*···O1*A*($-x+\frac{1}{2}, y+\frac{1}{2}, -z+2$) and N11—H11*C*···O2*A*($-x+\frac{1}{2}, y-\frac{1}{2}, -z+2$) hydrogen bonds connecting 2_1-related amino acids (Vijayan, 1988). The O^γ atom of threoninium molecule *A*, as acceptor, links the carboxyl O atom of threoninium molecule *B* through a strong hydrogen bond, O2*B*—H2*B*···O1*C*($x, y+1, z$). The two water molecules, lying on the twofold axes, link (i) the sulfate groups

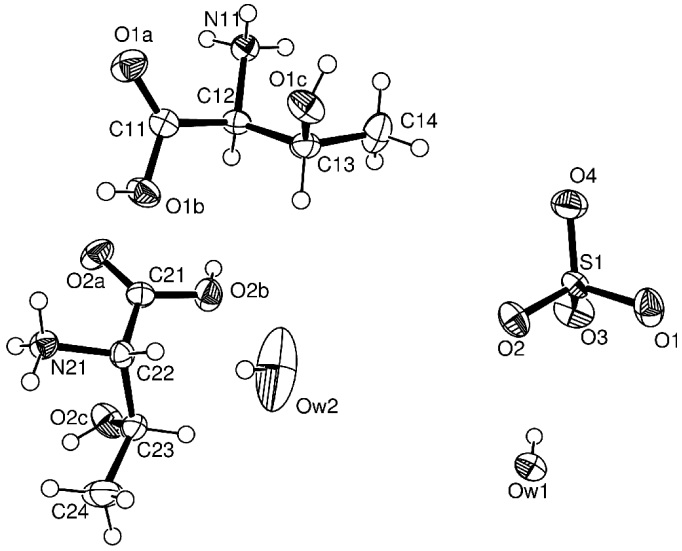


Figure 1
Views of the two independent threonine cations showing the atomic numbering scheme and 50% probability displacement ellipsoids (Johnson, 1976).

and (ii) threoninium molecule *B* through the O2*C* atom. One of the water molecules, as acceptor, links the amino group of threoninium molecule *B*, N21—H21*B*···O*W*1($x, y, 1 + z$). Two bifurcated hydrogen bonds are observed for the N21 amino group with sulfate O atoms through H21*A* and H21*C*. A four-centre hydrogen bond is observed in the case of N11—H11*C* involving the sulfate O atom and the double-bonded O atom of the carboxyl group of both molecules, connecting all the moieties in the structure (Jeffrey & Saenger, 1991). The sulfate anion links through the N11 atom of three threoninium *A* molecules, resulting in infinite chains along the *b* axis. The O3 atom of the sulfate anion links the N21 atoms of two threoninium *B* molecules, resulting in infinite chains along the *c* axis.

Experimental

Crystals of (I) were obtained from an aqueous solution of a 2:1 stoichiometric ratio of L-threonine and sulfuric acid.

Crystal data

$2C_4H_{10}NO_3^+\cdot SO_4^{2-}\cdot H_2O$
$M_r = 354.34$
Monoclinic, *C*2
$a = 23.096\ (4)$ Å
$b = 6.281\ (9)$ Å
$c = 11.648\ (1)$ Å
$\beta = 116.122\ (9)^\circ$
$V = 1517\ (2)$ Å^3
$Z = 4$
$D_x = 1.551$ Mg m^{-3}
$D_m = 1.54$ Mg m^{-3}
D_m measured by flotation in carbon tetrachloride and xylene
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.8$–14.4°
$\mu = 0.27$ mm^{-1}
$T = 293\ (2)$ K
Needle, colorless
$0.50 \times 0.45 \times 0.33$ mm

Data collection

Enraf–Nonius sealed-tube diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.869$, $T_{max} = 0.914$
1872 measured reflections
1732 independent reflections
1675 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 25.0^\circ$
$h = -1 \rightarrow 27$
$k = -1 \rightarrow 7$
$l = -13 \rightarrow 12$
25 standard reflections
every 3 reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.094$
$S = 0.98$
1732 reflections
213 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0764P)^2 + 1.0496P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.026 (2)
Absolute structure: (Flack, 1983)
Flack parameter = −0.07 (10)

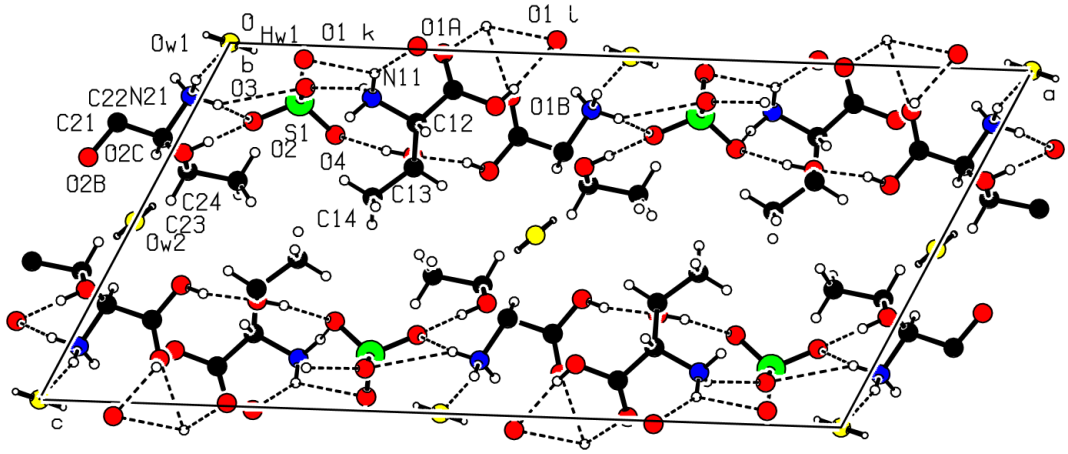


Figure 2
Packing diagram of the molecule viewed down the *b* axis.

Table 1
Selected torsion angles (°).

O1*A*—C11—C12—N11	−12.6 (4)	O2*A*—C21—C22—N21	−8.3 (4)
N11—C12—C13—O1*C*	75.8 (3)	N21—C22—C23—O2*C*	78.6 (3)
N11—C12—C13—C14	−49.2 (4)	N21—C22—C23—C24	−48.1 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O*1B*—H1*B*···O1^{i}	0.82	1.89	2.666 (3)	157
N11—H11*A*···O4^{ii}	0.89	2.30	2.927 (5)	128
N11—H11*B*···O3^{iii}	0.89	2.04	2.761 (4)	137
N11—H11*C*···O1*A*^{iv}	0.89	2.10	2.846 (4)	141
N11—H11*C*···O2*A*^{v}	0.89	2.33	2.837 (3)	117
N11—H11*C*···O1^{ii}	0.89	2.58	3.052 (4)	114
O1*C*—H1*C*···O4^{iii}	0.82	1.97	2.746 (3)	157
O2*B*—H2*B*···O1*C*^{vi}	0.82	1.79	2.600 (3)	167
N21—H21*A*···O2^{vii}	0.89	2.01	2.797 (4)	148
N21—H21*A*···O3^{vii}	0.89	2.57	3.337 (3)	144
N21—H21*B*···O*W*1^{i}	0.89	1.92	2.764 (4)	157
N21—H21*C*···O3^{i}	0.89	2.39	3.038 (3)	130
N21—H21*C*···O1^{i}	0.89	2.62	3.477 (5)	161
O2C—H2*C*···O2^{viii}	0.82	2.02	2.764 (3)	151
O*W*1—H*W*1···O1^{vi}	0.81 (5)	1.93 (5)	2.719 (3)	165 (6)
O*W*2—H*W*2···O2*C*^{ix}	0.86 (7)	2.07 (7)	2.908 (4)	162 (7)

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

The H atoms of the water molecules were located by difference Fourier maps and were refined, while all other H atoms were fixed with geometric restraints using *HFIX* and allowed to ride on the parent atom.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97.

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