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Tris(diisopropylammonium) hydrogensulfate sulfate

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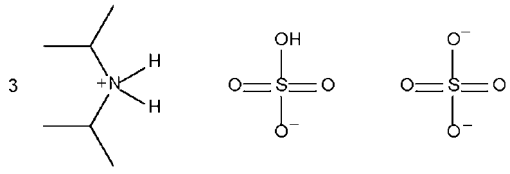
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.050; wR factor = 0.122; data-to-parameter ratio = 18.8.

The cations and anions of the title salt, $3C_6H_{16}N^+\cdot HSO_4^-\cdot SO_4^{2-}$, are linked by N—H···O and O—H···O hydrogen bonds into a three-dimensional network. The hydrogensulfate ion, with a single S—O(H) bond of 1.563 (2) Å, forms a short O—H···O hydrogen bond [O···O = 2.609 (2) Å] to the sulfate ion. The hydrogensulfate ion accepts two hydrogen bonds from two cations, whereas the sulfate ion, as an acceptor, binds to four cations. The sulfate ion is disordered approximately equally over two sites related by rotation around one of the O—S bonds.

Related literature

For the crystal structures of other hydrogensulfate–sulfate salts, see: Anderson *et al.* (2006); Banerjee & Murugavel (2004); Kang *et al.* (2005); Novozhilova *et al.* (1987); Sridhar *et al.* (2001); Warden *et al.* (2004). For the synthesis of ammonium sulfates, see: Jordanovska *et al.* (2000).



Experimental

Crystal data

$3C_6H_{16}N^+\cdot HSO_4^-\cdot SO_4^{2-}$
M_r = 499.72
Monoclinic, $P2_1/c$
a = 8.6178 (6) Å
b = 16.741 (1) Å
c = 19.819 (1) Å
β = 101.973 (5)°
V = 2797.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 295 (2) K
0.40 × 0.30 × 0.25 mm

Data collection

Stoe IPDSII imaging plate diffractometer
Absorption correction: analytical (*X-SHAPE*; Stoe & Cie, 2003)
T_{min} = 0.91, T_{max} = 0.94
16154 measured reflections
6311 independent reflections
4905 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.122
S = 1.06
6311 reflections
336 parameters
94 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7···O4	0.86 (1)	1.76 (1)	2.609 (2)	174 (4)
N1—H1*N*1···O1	0.86 (1)	1.76 (1)	2.585 (5)	159 (3)
N1—H1*N*2···O6^{i}	0.85 (1)	2.02 (1)	2.874 (2)	176 (3)
N2—H2*N*2···O4	0.85 (1)	2.10 (1)	2.929 (2)	166 (2)
N2—H2*N*1···O2^{ii}	0.85 (1)	1.85 (1)	2.695 (6)	179 (3)
N2—H2*N*1···O2′^{ii}	0.85 (1)	2.02 (2)	2.855 (8)	166 (3)
N3—H3*N*1···O3	0.86 (1)	1.89 (1)	2.738 (8)	174 (2)
N3—H3*N*1···O3′	0.86 (1)	1.88 (1)	2.711 (7)	162 (2)
N3—H3*N*2···O5^{iii}	0.86 (1)	2.00 (1)	2.819 (2)	159 (2)

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

Data collection: *X-RED* (Stoe & Cie, 2001); cell refinement: *X-AREA* (Stoe & Cie, 2005); data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2158).

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supplementary materials

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Tris(diisopropylammonium) hydrogensulfate sulfate

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Comment

Disubstituted ammonium sulfates are used in the synthesis of double salts with other metal sulfates (Jordanovska *et al.*, 2000). The reaction of diisopropylamine with sulfuric acid yielded the expected compound as a double sulfate with diisopropylammonium hydrogensulfate (Fig. 1). A small number of such double salts are known (Anderson *et al.*, 2006; Banerjee & Murugavel, 2004; Kang *et al.*, 2005; Novozhilova *et al.*, 1987; Sridhar *et al.*, 2001; Warden *et al.*, 2004). In the title compound, the cations and anions are linked by N–H···O and O–H···O hydrogen bonds into three-dimensional network structure. The sulfate ion is disordered over two sites in an "umbrella" type of disorder (only three of the four oxygen atoms are disordered).

Experimental

Following the method of Jordanovska *et al.* (2000), diisopropylamine (1 ml, 7.1 mmol) was dissolved in chloroform (10 ml) and concentrated sulfuric acid was added dropwise at 273 K until a white precipitate was formed. The precipitate was collected and recrystallized from water.

Refinement

The sulfate ion is disordered over two positions related by rotation around the S1-O4 bond. For this ion, all S–O distances were restrained to be equal within 0.01 Å. Similar restraints were were imposed on O···O distances within this ion.

Carbon-bound hydrogen atoms were placed in calculated positions (C–H 0.96 – 0.98 Å), and were included in refinement in the riding model approximation, with U(H) set to 1.2-1.5 times U_{eq}(C). Oxygen and nitrogen-bound hydrogen atoms were located in a difference Fourier map, and were refined with a distance restraint (O,N)–H= 0.85±0.01 Å; their temperature factors were freely refined.

Figures

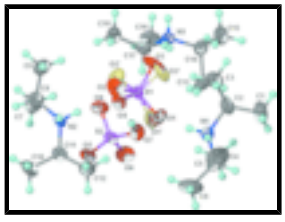


Fig. 1. Molecular structure of $[(C_6H_{16}N)_3]_3$ [HSO4] [SO4]; displacement ellipsoids are drawn at the 50% probablity level and H atoms are drawn as spheres of arbitrary radii. Disorder in the sulfate ion is shown.

Tris(diisopropylammonium) hydrogensulfate sulfate

Crystal data

$3C_6H_{16}N^+ \cdot HSO_4^- \cdot SO_4^{2-}$	$F_{000} = 1096$
$M_r = 499.72$	$D_x = 1.187$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 4365 reflections
$a = 8.6178$ (6) Å	$\theta = 2.4$–27.5º
$b = 16.741$ (1) Å	$\mu = 0.23$ mm^{-1}
$c = 19.819$ (1) Å	$T = 295$ (2) K
$\beta = 101.973$ (5)º	Block, colorless
$V = 2797.2$ (3) Å^3	0.40 × 0.30 × 0.25 mm
$Z = 4$	

Data collection

Stoe IPDS-II imaging plate diffractometer	6311 independent reflections
Radiation source: medium-focus sealed tube	4905 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.035$
$T = 295$(2) K	$\theta_{max} = 27.5$º
Rotation method scans	$\theta_{min} = 2.4$º
Absorption correction: analytical (X-SHAPE; Stoe & Cie, 2003)	$h = -11 \rightarrow 11$
$T_{min} = 0.91$, $T_{max} = 0.94$	$k = -21 \rightarrow 21$
16154 measured reflections	$l = -25 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.049$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.122$	$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.953P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
6311 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
336 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
94 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.69023 (5)	0.61641 (3)	0.59691 (2)	0.03414 (13)	
S2	0.50949 (5)	0.60829 (3)	0.81272 (3)	0.03843 (14)	
O1	0.6113 (17)	0.6922 (6)	0.5791 (8)	0.093 (4)	0.49 (3)
O2	0.6440 (16)	0.5588 (7)	0.5417 (4)	0.068 (3)	0.49 (3)
O3	0.8606 (6)	0.6263 (9)	0.6126 (4)	0.072 (3)	0.49 (3)
O4	0.63401 (18)	0.58408 (10)	0.65737 (8)	0.0505 (4)	
O1'	0.5800 (8)	0.6754 (5)	0.5608 (5)	0.082 (3)	0.51 (3)
O2'	0.6958 (17)	0.5496 (4)	0.5505 (4)	0.065 (2)	0.51 (3)
O3'	0.8453 (7)	0.6519 (7)	0.6185 (4)	0.068 (2)	0.51 (3)
O5	0.36497 (17)	0.57133 (11)	0.77530 (9)	0.0522 (4)	
O6	0.4807 (2)	0.66251 (12)	0.86590 (9)	0.0640 (5)	
O7	0.5682 (2)	0.66486 (10)	0.76049 (10)	0.0562 (4)	
H7	0.584 (4)	0.640 (2)	0.7247 (12)	0.099 (12)*	
O8	0.63320 (19)	0.55114 (12)	0.83566 (10)	0.0625 (5)	
N1	0.6123 (2)	0.82382 (11)	0.51105 (9)	0.0410 (4)	
H1n1	0.604 (3)	0.7752 (8)	0.5238 (13)	0.046 (6)*	
H1n2	0.577 (3)	0.8265 (16)	0.4676 (6)	0.057 (7)*	
N2	0.4034 (2)	0.46240 (11)	0.59581 (9)	0.0378 (4)	
H2n1	0.390 (3)	0.4557 (16)	0.5525 (6)	0.059 (8)*	
H2n2	0.480 (2)	0.4947 (12)	0.6089 (13)	0.049 (7)*	
N3	1.0587 (2)	0.63815 (11)	0.73947 (9)	0.0379 (4)	
H3n1	1.003 (2)	0.6345 (14)	0.6985 (7)	0.047 (7)*	
H3n2	1.1482 (17)	0.6160 (13)	0.7392 (13)	0.044 (6)*	
C1	0.8175 (4)	0.92758 (18)	0.50748 (18)	0.0813 (9)	
H1A	0.7674	0.9628	0.5347	0.122*	
H1B	0.7749	0.9369	0.4594	0.122*	
H1C	0.9297	0.9374	0.5172	0.122*	
C2	0.7866 (3)	0.84177 (15)	0.52506 (12)	0.0518 (6)	
H2	0.8301	0.8333	0.5743	0.062*	
C3	0.8635 (3)	0.78284 (19)	0.48440 (18)	0.0711 (8)	
H3A	0.8421	0.7294	0.4975	0.107*	
H3B	0.9761	0.7916	0.4938	0.107*	
H3C	0.8213	0.7900	0.4360	0.107*	
C4	0.5666 (5)	0.8766 (2)	0.62260 (17)	0.0905 (11)	
H4A	0.6716	0.8988	0.6330	0.136*	
H4B	0.5689	0.8234	0.6408	0.136*	
H4C	0.4970	0.9091	0.6431	0.136*	
C5	0.5075 (3)	0.87439 (16)	0.54560 (14)	0.0609 (7)	
H5	0.5067	0.9290	0.5277	0.073*	
C6	0.3412 (4)	0.8405 (3)	0.5257 (2)	0.0934 (12)	
H6A	0.3080	0.8401	0.4763	0.140*	
H6B	0.2698	0.8729	0.5451	0.140*	
H6C	0.3404	0.7869	0.5429	0.140*	
C7	0.4706 (4)	0.38395 (19)	0.70440 (14)	0.0758 (9)	
H7A	0.3691	0.3969	0.7143	0.114*	

H7B	0.5470	0.4233	0.7248	0.114*
H7C	0.5035	0.3323	0.7231	0.114*
C8	0.4583 (3)	0.38282 (13)	0.62715 (12)	0.0459 (5)
H8	0.3798	0.3424	0.6072	0.055*
C9	0.6128 (3)	0.36272 (16)	0.60696 (15)	0.0620 (7)
H9A	0.5979	0.3624	0.5576	0.093*
H9B	0.6479	0.3110	0.6247	0.093*
H9C	0.6911	0.4020	0.6257	0.093*
C10	0.1143 (3)	0.44046 (19)	0.58522 (16)	0.0659 (7)
H10A	0.1346	0.3909	0.6098	0.099*
H10B	0.1027	0.4309	0.5367	0.099*
H10C	0.0185	0.4637	0.5939	0.099*
C11	0.2517 (3)	0.49718 (14)	0.60939 (12)	0.0461 (5)
H11	0.2637	0.5058	0.6591	0.055*
C12	0.2265 (3)	0.57729 (16)	0.57311 (16)	0.0597 (6)
H12A	0.3155	0.6114	0.5903	0.090*
H12B	0.1316	0.6015	0.5817	0.090*
H12C	0.2166	0.5697	0.5244	0.090*
C13	0.9325 (4)	0.76767 (18)	0.7572 (2)	0.0849 (10)
H13A	0.8896	0.7448	0.7938	0.127*
H13B	0.8580	0.7612	0.7142	0.127*
H13C	0.9526	0.8235	0.7660	0.127*
C14	1.0867 (3)	0.72570 (14)	0.75319 (14)	0.0547 (6)
H14	1.1609	0.7320	0.7976	0.066*
C15	1.1633 (4)	0.75867 (18)	0.69695 (19)	0.0760 (9)
H15A	1.2604	0.7306	0.6970	0.114*
H15B	1.1854	0.8144	0.7051	0.114*
H15C	1.0926	0.7519	0.6530	0.114*
C16	1.0647 (3)	0.5999 (2)	0.86078 (14)	0.0712 (8)
H16A	1.0666	0.6552	0.8738	0.107*
H16B	1.1714	0.5808	0.8655	0.107*
H16C	1.0113	0.5694	0.8901	0.107*
C17	0.9777 (2)	0.59106 (15)	0.78665 (13)	0.0476 (5)
H17	0.8696	0.6116	0.7824	0.057*
C18	0.9683 (3)	0.50527 (16)	0.76210 (19)	0.0720 (8)
H18A	0.9115	0.5029	0.7150	0.108*
H18B	0.9139	0.4738	0.7904	0.108*
H18C	1.0735	0.4847	0.7653	0.108*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0348 (2)	0.0397 (3)	0.0275 (2)	−0.00589 (19)	0.00538 (17)	0.00145 (19)
S2	0.0328 (2)	0.0516 (3)	0.0309 (2)	0.0025 (2)	0.00650 (18)	−0.0062 (2)
O1	0.102 (6)	0.046 (3)	0.155 (7)	0.025 (4)	0.081 (5)	0.045 (4)
O2	0.077 (5)	0.100 (5)	0.028 (2)	−0.031 (4)	0.011 (3)	−0.018 (3)
O3	0.029 (2)	0.140 (8)	0.046 (3)	−0.017 (3)	0.0059 (19)	−0.009 (4)
O4	0.0605 (9)	0.0598 (10)	0.0357 (8)	−0.0112 (8)	0.0203 (7)	−0.0017 (7)

O1'	0.050 (3)	0.056 (3)	0.127 (5)	−0.015 (3)	−0.011 (4)	0.045 (3)
O2'	0.100 (6)	0.051 (3)	0.044 (3)	0.002 (3)	0.014 (3)	−0.011 (2)
O3'	0.057 (3)	0.096 (5)	0.045 (3)	−0.040 (3)	−0.008 (2)	0.025 (3)
O5	0.0340 (7)	0.0676 (11)	0.0536 (10)	−0.0023 (7)	0.0058 (6)	−0.0119 (8)
O6	0.0698 (11)	0.0832 (13)	0.0400 (9)	0.0056 (10)	0.0138 (8)	−0.0220 (9)
O7	0.0715 (11)	0.0512 (10)	0.0520 (10)	−0.0055 (8)	0.0265 (9)	−0.0041 (8)
O8	0.0450 (9)	0.0744 (12)	0.0647 (12)	0.0125 (8)	0.0037 (8)	0.0073 (9)
N1	0.0502 (10)	0.0411 (10)	0.0312 (9)	−0.0023 (8)	0.0068 (7)	0.0063 (7)
N2	0.0391 (9)	0.0437 (10)	0.0297 (9)	−0.0076 (7)	0.0046 (7)	0.0014 (7)
N3	0.0317 (8)	0.0428 (9)	0.0365 (9)	0.0022 (7)	0.0010 (7)	−0.0008 (7)
C1	0.099 (2)	0.0678 (19)	0.076 (2)	−0.0386 (17)	0.0171 (17)	−0.0039 (16)
C2	0.0525 (12)	0.0613 (14)	0.0389 (12)	−0.0140 (11)	0.0031 (9)	0.0054 (10)
C3	0.0594 (15)	0.0784 (19)	0.083 (2)	−0.0034 (14)	0.0311 (14)	0.0107 (16)
C4	0.120 (3)	0.101 (3)	0.0549 (18)	0.025 (2)	0.0283 (18)	−0.0156 (17)
C5	0.0791 (18)	0.0545 (14)	0.0517 (15)	0.0212 (13)	0.0196 (12)	0.0102 (11)
C6	0.0603 (17)	0.137 (3)	0.087 (2)	0.031 (2)	0.0243 (16)	0.030 (2)
C7	0.114 (2)	0.0731 (19)	0.0387 (14)	0.0098 (17)	0.0132 (14)	0.0174 (13)
C8	0.0604 (13)	0.0373 (11)	0.0375 (11)	−0.0082 (10)	0.0044 (9)	0.0023 (9)
C9	0.0673 (16)	0.0542 (14)	0.0614 (16)	0.0126 (12)	0.0063 (13)	0.0080 (12)
C10	0.0451 (13)	0.0831 (19)	0.0718 (19)	−0.0161 (13)	0.0175 (12)	−0.0057 (15)
C11	0.0446 (11)	0.0612 (14)	0.0337 (11)	−0.0034 (10)	0.0109 (9)	−0.0096 (10)
C12	0.0482 (13)	0.0610 (15)	0.0682 (17)	0.0057 (11)	0.0081 (11)	−0.0031 (13)
C13	0.088 (2)	0.0484 (15)	0.122 (3)	0.0157 (15)	0.030 (2)	−0.0075 (17)
C14	0.0540 (13)	0.0440 (12)	0.0603 (15)	−0.0026 (10)	−0.0017 (11)	−0.0095 (11)
C15	0.0704 (18)	0.0574 (17)	0.098 (2)	−0.0149 (14)	0.0111 (16)	0.0124 (16)
C16	0.0617 (15)	0.109 (2)	0.0455 (14)	0.0151 (15)	0.0178 (12)	0.0147 (15)
C17	0.0341 (10)	0.0587 (13)	0.0511 (13)	0.0062 (9)	0.0114 (9)	0.0079 (10)
C18	0.0656 (17)	0.0508 (15)	0.100 (3)	−0.0043 (13)	0.0192 (16)	0.0134 (15)

Geometric parameters (Å, °)

S1—O3	1.445 (4)	C6—H6A	0.9600
S1—O3'	1.444 (4)	C6—H6B	0.9600
S1—O1	1.448 (4)	C6—H6C	0.9600
S1—O2	1.450 (5)	C7—C8	1.513 (3)
S1—O1'	1.452 (4)	C7—H7A	0.9600
S1—O2'	1.456 (4)	C7—H7B	0.9600
S1—O4	1.4848 (15)	C7—H7C	0.9600
S2—O8	1.4344 (17)	C8—C9	1.506 (4)
S2—O5	1.4494 (16)	C8—H8	0.9800
S2—O6	1.4508 (17)	C9—H9A	0.9600
S2—O7	1.5627 (18)	C9—H9B	0.9600
O7—H7	0.856 (10)	C9—H9C	0.9600
N1—C5	1.502 (3)	C10—C11	1.516 (3)
N1—C2	1.500 (3)	C10—H10A	0.9600
N1—H1n1	0.860 (10)	C10—H10B	0.9600
N1—H1n2	0.854 (10)	C10—H10C	0.9600
N2—C8	1.504 (3)	C11—C12	1.516 (4)
N2—C11	1.506 (3)	C11—H11	0.9800

N2—H2n1	0.849 (10)	C12—H12A	0.9600
N2—H2n2	0.851 (10)	C12—H12B	0.9600
N3—C17	1.501 (3)	C12—H12C	0.9600
N3—C14	1.501 (3)	C13—C14	1.519 (4)
N3—H3n1	0.856 (10)	C13—H13A	0.9600
N3—H3n2	0.857 (10)	C13—H13B	0.9600
C1—C2	1.515 (4)	C13—H13C	0.9600
C1—H1A	0.9600	C14—C15	1.513 (4)
C1—H1B	0.9600	C14—H14	0.9800
C1—H1C	0.9600	C15—H15A	0.9600
C2—C3	1.511 (4)	C15—H15B	0.9600
C2—H2	0.9800	C15—H15C	0.9600
C3—H3A	0.9600	C16—C17	1.512 (4)
C3—H3B	0.9600	C16—H16A	0.9600
C3—H3C	0.9600	C16—H16B	0.9600
C4—C5	1.506 (4)	C16—H16C	0.9600
C4—H4A	0.9600	C17—C18	1.513 (4)
C4—H4B	0.9600	C17—H17	0.9800
C4—H4C	0.9600	C18—H18A	0.9600
C5—C6	1.516 (5)	C18—H18B	0.9600
C5—H5	0.9800	C18—H18C	0.9600
O3—S1—O1	110.9 (4)	C8—C7—H7A	109.5
O3—S1—O2	110.3 (4)	C8—C7—H7B	109.5
O1—S1—O2	110.9 (4)	H7A—C7—H7B	109.5
O3'—S1—O1'	109.7 (4)	C8—C7—H7C	109.5
O3'—S1—O2'	110.7 (4)	H7A—C7—H7C	109.5
O1'—S1—O2'	108.3 (4)	H7B—C7—H7C	109.5
O3—S1—O4	110.8 (4)	C9—C8—N2	107.92 (19)
O3'—S1—O4	110.3 (3)	C9—C8—C7	113.1 (2)
O1—S1—O4	107.5 (3)	N2—C8—C7	110.9 (2)
O2—S1—O4	106.4 (3)	C9—C8—H8	108.3
O1'—S1—O4	110.9 (3)	N2—C8—H8	108.3
O2'—S1—O4	106.8 (3)	C7—C8—H8	108.3
O8—S2—O5	112.37 (11)	C8—C9—H9A	109.5
O8—S2—O6	114.51 (11)	C8—C9—H9B	109.5
O5—S2—O6	112.38 (10)	H9A—C9—H9B	109.5
O8—S2—O7	107.02 (11)	C8—C9—H9C	109.5
O5—S2—O7	106.37 (10)	H9A—C9—H9C	109.5
O6—S2—O7	103.28 (11)	H9B—C9—H9C	109.5
S2—O7—H7	112 (3)	C11—C10—H10A	109.5
C5—N1—C2	118.4 (2)	C11—C10—H10B	109.5
C5—N1—H1n1	107.6 (17)	H10A—C10—H10B	109.5
C2—N1—H1n1	106.2 (16)	C11—C10—H10C	109.5
C5—N1—H1n2	107.9 (18)	H10A—C10—H10C	109.5
C2—N1—H1n2	108.1 (18)	H10B—C10—H10C	109.5
H1n1—N1—H1n2	108 (2)	N2—C11—C10	110.6 (2)
C8—N2—C11	118.61 (18)	N2—C11—C12	107.53 (19)
C8—N2—H2n1	105.4 (18)	C10—C11—C12	112.3 (2)
C11—N2—H2n1	106.8 (18)	N2—C11—H11	108.8

C8—N2—H2n2	106.2 (17)	C10—C11—H11	108.8
C11—N2—H2n2	110.3 (17)	C12—C11—H11	108.8
H2n1—N2—H2n2	109 (3)	C11—C12—H12A	109.5
C17—N3—C14	118.56 (19)	C11—C12—H12B	109.5
C17—N3—H3n1	108.2 (17)	H12A—C12—H12B	109.5
C14—N3—H3n1	106.2 (16)	C11—C12—H12C	109.5
C17—N3—H3n2	108.2 (17)	H12A—C12—H12C	109.5
C14—N3—H3n2	108.2 (16)	H12B—C12—H12C	109.5
H3n1—N3—H3n2	107 (2)	C14—C13—H13A	109.5
C2—C1—H1A	109.5	C14—C13—H13B	109.5
C2—C1—H1B	109.5	H13A—C13—H13B	109.5
H1A—C1—H1B	109.5	C14—C13—H13C	109.5
C2—C1—H1C	109.5	H13A—C13—H13C	109.5
H1A—C1—H1C	109.5	H13B—C13—H13C	109.5
H1B—C1—H1C	109.5	N3—C14—C15	107.5 (2)
N1—C2—C3	107.7 (2)	N3—C14—C13	110.6 (2)
N1—C2—C1	111.4 (2)	C15—C14—C13	112.9 (3)
C3—C2—C1	112.3 (2)	N3—C14—H14	108.6
N1—C2—H2	108.5	C15—C14—H14	108.6
C3—C2—H2	108.5	C13—C14—H14	108.6
C1—C2—H2	108.5	C14—C15—H15A	109.5
C2—C3—H3A	109.5	C14—C15—H15B	109.5
C2—C3—H3B	109.5	H15A—C15—H15B	109.5
H3A—C3—H3B	109.5	C14—C15—H15C	109.5
C2—C3—H3C	109.5	H15A—C15—H15C	109.5
H3A—C3—H3C	109.5	H15B—C15—H15C	109.5
H3B—C3—H3C	109.5	C17—C16—H16A	109.5
C5—C4—H4A	109.5	C17—C16—H16B	109.5
C5—C4—H4B	109.5	H16A—C16—H16B	109.5
H4A—C4—H4B	109.5	C17—C16—H16C	109.5
C5—C4—H4C	109.5	H16A—C16—H16C	109.5
H4A—C4—H4C	109.5	H16B—C16—H16C	109.5
H4B—C4—H4C	109.5	N3—C17—C16	110.7 (2)
N1—C5—C4	111.5 (2)	N3—C17—C18	107.4 (2)
N1—C5—C6	107.2 (2)	C16—C17—C18	112.8 (2)
C4—C5—C6	112.1 (3)	N3—C17—H17	108.6
N1—C5—H5	108.6	C16—C17—H17	108.6
C4—C5—H5	108.6	C18—C17—H17	108.6
C6—C5—H5	108.6	C17—C18—H18A	109.5
C5—C6—H6A	109.5	C17—C18—H18B	109.5
C5—C6—H6B	109.5	H18A—C18—H18B	109.5
H6A—C6—H6B	109.5	C17—C18—H18C	109.5
C5—C6—H6C	109.5	H18A—C18—H18C	109.5
H6A—C6—H6C	109.5	H18B—C18—H18C	109.5
H6B—C6—H6C	109.5		
C5—N1—C2—C3	−180.0 (2)	C8—N2—C11—C10	−58.1 (3)
C5—N1—C2—C1	−56.5 (3)	C8—N2—C11—C12	179.04 (18)
C2—N1—C5—C4	−53.9 (3)	C17—N3—C14—C15	−177.8 (2)
C2—N1—C5—C6	−176.9 (2)	C17—N3—C14—C13	−54.1 (3)

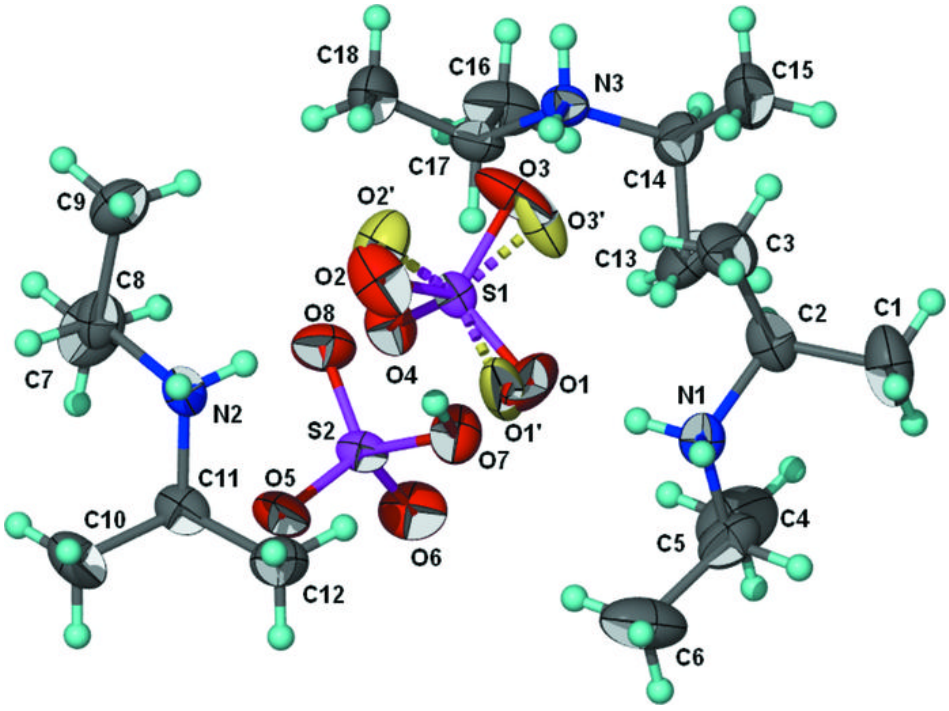
C11—N2—C8—C9	−178.90 (19)	C14—N3—C17—C16	−55.2 (3)
C11—N2—C8—C7	−54.6 (3)	C14—N3—C17—C18	−178.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7···O4	0.86 (1)	1.76 (1)	2.609 (2)	174 (4)
N1—H1N1···O1	0.86 (1)	1.76 (1)	2.585 (5)	159 (3)
N1—H1N2···O6^{i}	0.85 (1)	2.02 (1)	2.874 (2)	176 (3)
N2—H2N2···O4	0.85 (1)	2.10 (1)	2.929 (2)	166 (2)
N2—H2N1···O2^{ii}	0.85 (1)	1.85 (1)	2.695 (6)	179 (3)
N2—H2N1···O2^{iii}	0.85 (1)	2.02 (2)	2.855 (8)	166 (3)
N3—H3N1···O3	0.86 (1)	1.89 (1)	2.738 (8)	174 (2)
N3—H3N1···O3'	0.86 (1)	1.88 (1)	2.711 (7)	162 (2)
N3—H3N2···O5^{iii}	0.86 (1)	2.00 (1)	2.819 (2)	159 (2)

Symmetry codes: (i) *x*, −*y*+3/2, *z*−1/2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*+1, *y*, *z*.

Fig. 1


C18
C16
N3
C15
C17
O3
C14
O2'
O3'
C9
C13
C3
O2
S1
C8
C2
C1
O8
O4
O1
C7
N1
N2
S2
O1'
O7
C11
O5
C4
C10
C5
O6
C12
C6