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Bis[diethylenetriaminium(3+)] tris(sulfate) monohydrate

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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 = 0.058
wR factor = 0.154
Data-to-parameter ratio = 15.9

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

The cations, anions and water molecules in the title compound, $2C_4H_{16}N_3^{3+}\cdot 3SO_4^{2-}\cdot H_2O$, are linked by hydrogen bonds into a three-dimensional network structure. The water molecule and one sulfate anion lie on special positions of site symmetry 2; one of the sulfate anions is disordered.

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Comment

The diethylenetriammonium $[NH_3CH_2CH_2NH_2CH_2CH_2NH_3]$ trication forms only a small number of salts with mineral acids. The trication has been crystallographically characterized as the trichloride (Golubev & Kondrashev, 1981; Ilioudis *et al.*, 2000), tribromide (Ilioudis *et al.*, 2000), chloride diperchlorate (Mazus *et al.*, 1987), trinitrate (Rogers & Bauer, 1994) and cyclophosphate dihydrate (Gharbi *et al.*, 1995). The title compound exists as a monohydrate, (I) (Fig. 1); there are two independent sulfate dianions, one of which is disordered about a twofold axis. Hydrogen bonds link the cations, anions and water molecules into a three-dimensional network structure (Table 2).

$$2[NH_3CH_2CH_2NH_2CH_2CH_2NH_3]^{3+}\ 3[SO_4]^{2-}\ H_2O$$
(I)

Experimental

Ferric sulfate nonahydrate (0.281 g, 0.5 mmol), sodium acetate (0.21 g, 1 mmol), diethylenetriamine (0.15 ml, 0.13 mmol), concen-

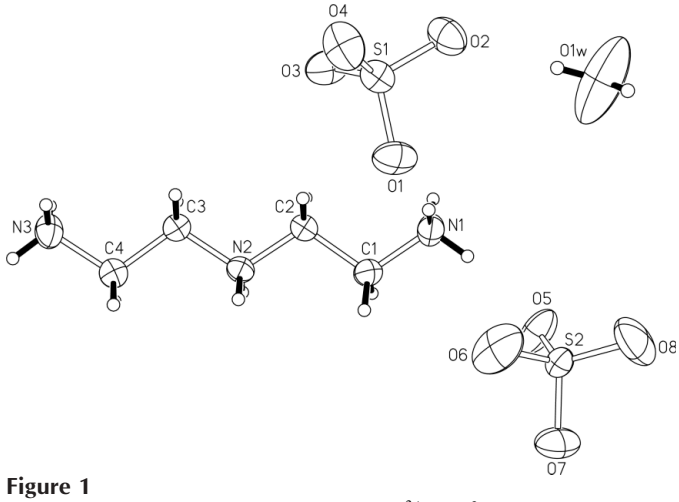

Figure 1
ORTEPII (Johnson, 1976) plot of $2C_4H_{16}N_3^{3+}\cdot 3SO_4^{2-}\cdot H_2O$. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii. Only one set of the disordered O atoms of S2 is shown.

trated sulfuric acid (0.16 ml), water (6 ml), ethanol (5 ml) and glycol (7 ml) were placed in a Teflon-lined stainless steel bomb. The bomb was heated in an autoclave at 383 K for 4 d and then cooled to room temperature to furnish crystals of (I). Iron was not incorporated into the compound isolated.

Crystal data

$2C_4H_{16}N_3^{3+}\cdot 3SO_4^{2-}\cdot H_2O$
$M_r = 518.59$
Monoclinic, $C2/c$
$a = 10.249$ (1) Å
$b = 13.648$ (1) Å
$c = 16.082$ (2) Å
$\beta = 102.045$ (2)°
$V = 2199.9$ (4) Å^3
$Z = 4$
$D_x = 1.566$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1515 reflections
$\theta = 2.5$–27.1°
$\mu = 0.41$ mm^{-1}
$T = 295$ (2) K
Block, yellow
0.15 × 0.13 × 0.12 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: None
6500 measured reflections
2468 independent reflections
1971 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 13$
$k = -17 \rightarrow 17$
$l = -16 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.154$
$S = 1.02$
2468 reflections
155 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0814P)^2 + 3.1409P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Extinction correction: none

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.476 (2)	S2—O8	1.439 (4)
S1—O2	1.470 (2)	N1—C1	1.474 (4)
S1—O3	1.473 (2)	N2—C3	1.474 (4)
S1—O4	1.455 (2)	N2—C2	1.474 (4)
S2—O5	1.471 (3)	N3—C4	1.466 (4)
S2—O6	1.465 (4)	C1—C2	1.510 (4)
S2—O7	1.486 (3)	C3—C4	1.500 (4)
O1—S1—O2	108.6 (2)	O6—S2—O7	104.5 (4)
O1—S1—O3	109.1 (1)	O6—S2—O8	115.5 (4)
O1—S1—O4	109.2 (2)	O7—S2—O8	109.9 (4)
O2—S1—O3	107.4 (2)	C3—N2—C2	113.0 (2)
O2—S1—O4	110.8 (1)	N1—C1—C2	109.9 (2)
O3—S1—O4	111.7 (2)	N2—C2—C1	111.5 (2)
O5—S2—O6	108.6 (3)	N2—C3—C4	110.4 (2)
O5—S2—O7	103.1 (3)	N3—C4—C3	110.7 (3)
O5—S2—O8	114.1 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1w—H1w1···O2	0.86	2.03	2.875 (4)	169
N1—H1n2···O1	0.86	1.93	2.772 (4)	164
N1—H1n1···O5	0.86	1.95	2.793 (5)	166
N1—H1n3···O3^{i}	0.86	2.17	2.946 (4)	151
N2—H2n2···O1^{ii}	0.86	2.07	2.833 (3)	148
N2—H2n1···O8^{iii}	0.86	1.96	2.788 (6)	161
N3—H3n1···O2^{iv}	0.86	1.91	2.738 (4)	162
N3—H3n3···O6^{v}	0.86	2.22	3.001 (7)	151
N3—H3n3···O7^{v}	0.86	2.32	3.080 (6)	147
N3—H3n3···O8^{ii}	0.86	2.35	3.122 (8)	150
N3—H3n2···O3^{vi}	0.86	1.90	2.755 (4)	174

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

The sulfate group lying on the twofold axis is disordered, and the O atoms were refined as four O atoms of 0.5 site occupancy each. The four S—O distances were restrained to within 0.005 Å of each other. The C- and N-bound H atoms were placed at calculated positions (C—H = 0.97 Å and N—H = 0.86 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) = $1.2U_{eq}$(C,N). The water H atom was placed in a chemically sensible position on the basis of a hydrogen-bonding interaction and refined as riding [U_{iso}(H) = $1.2U_{eq}$(O)].

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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