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Triethylenetetraminium(4+) bis(sulfate) monohydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.070
wR factor = 0.185
Data-to-parameter ratio = 8.4

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

The cation and anion in the title salt, $C_6H_{22}N_4^{4+}\cdot 2SO_4^{2-}\cdot H_2O$, lie on special positions of site symmetry m and the water molecule on a special position of site symmetry $2/m$. The cations, anions and water molecules are linked by hydrogen bonds into a three-dimensional network.

Comment

The triethylenetetraammonium cation has been crystallographically authenticated in salts of some mineral acids. It forms a dihydrate with sulfuric acid when the parent amine is reacted with sulfuric acid (Ilioudis *et al.*, 2002). The title monohydrate, (I) (Fig. 1), which was obtained indirectly by way of a hydrothermal synthesis, exists as a three-dimensional hydrogen-bonded network structure, owing to extensive hydrogen bonds among the cations, anions and water molecules (Table 1). The cation and anion lie on special positions of site symmetry m and the water molecule on a special position of site symmetry $2/m$.

$$[NH_3CH_2CH_2NH_2CH_2CH_2NH_2CH_2CH_2NH_3]^{4+}\ 2[SO_4]^{2-}\ H_2O$$

(I)

Experimental

Ferric sulfate nonahydrate (0.281 g, 0.5 mmol), triethylenetetraamine (0.29 ml, 2 mmol), water (15 ml) and ethanol (5 ml) were placed in a Teflon-lined stainless steel bomb. The bomb was heated in an autoclave at 383 K for 4 d. The bomb was then cooled to room temperature to furnish crystals of (I). Iron was not incorporated in the compound isolated.

Crystal data

$C_6H_{22}N_4^{4+}\cdot 2SO_4^{2-}\cdot H_2O$
M_r = 360.41
Orthorhombic, $Pnnm$
a = 6.3192 (8) Å
b = 22.253 (3) Å
c = 5.4903 (7) Å
V = 772.1 (2) Å^3
Z = 2
D_x = 1.550 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 861 reflections
θ = 3.4–23.5°
μ = 0.39 mm^{-1}
T = 295 (2) K
Block, yellow
0.14 × 0.14 × 0.13 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
3543 measured reflections
946 independent reflections
840 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.035
$\theta_{\rm max}$ = 27.5°
$h = -8 \rightarrow 3$
$k = -25 \rightarrow 28$
$l = -6 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.185$
$S = 1.14$
946 reflections
113 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0921P)^2 + 1.2465P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1w—H1w1$\cdots$O3^{ii}	0.85	2.29	2.94 (1)	134
O1w—H1w1$\cdots$O3^{iii}	0.85	2.12	2.94 (1)	163
O1w—H1w2$\cdots$O6	0.85	2.05	2.681 (7)	131
O1w—H1w2$\cdots$O6^{iv}	0.85	2.10	2.681 (7)	125
O1w—H1w1$\cdots$O6^{ii}	0.85	2.07	2.681 (7)	128
O1w—H1w1$\cdots$O6^{iii}	0.85	2.05	2.681 (7)	131
N1—H1n1$\cdots$O2^{v}	0.86	1.98	2.818 (8)	163
N1—H1n1$\cdots$O5^{v}	0.86	2.12	2.86 (2)	145
N1—H1n2$\cdots$O1	0.86	2.01	2.83 (2)	159
N1—H1n2$\cdots$O7^{iv}	0.86	1.88	2.65 (2)	148
N2—H2$n\cdots$O3^{vi}	0.86	1.97	2.75 (2)	150
N2—H2$n\cdots$O4^{vii}	0.86	1.81	2.58 (2)	149
N2—H2$n\cdots$O7^{vii}	0.86	2.38	3.10 (2)	141
N2—H2$n\cdots$O8^{vi}	0.86	2.06	2.86 (3)	156
N2—H2$n\cdots$O8^{vii}	0.86	2.18	2.98 (3)	155

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

The sulfate group lying on the mirror plane is disordered, and the O atoms were refined as eight O atoms of 0.5 site occupancy each. The four S—O distances were restrained to within 0.01 Å of each other, as were the O$\cdots$O distances. The ellipsoids were restrained to be nearly isotropic.

The carbon- and nitrogen-bound H atoms were placed in calculated positions (C—H = 0.97 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 O atom lies on a site of symmetry $2/m$; the two disordered H atoms were placed in chemically sensible positions (O—H = 0.85 and H$\cdots$H = 1.39 Å) with half-occupancy, so that one of them was able to form a hydrogen bond. They were not refined; their U_{iso} values were constrained to $1.2U_{eq}$(O).

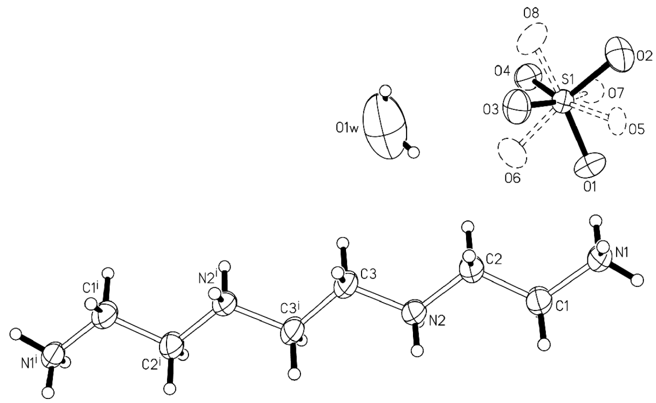

Figure 1
A plot of the three components of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The second disorder component (atoms O5, O6, O7 and O8) is shown as dotted ellipsoids and bonds. [Symmetry code (i): $\frac{3}{2}-x, \frac{1}{2}+y, z-\frac{1}{2}$.]

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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