METAL-ORGANIC COMPOUNDS

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Manganese Ethylenediammonium Bis(sulfate) Tetrahydrate

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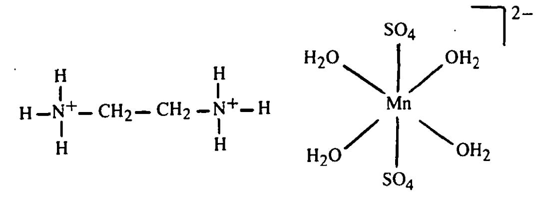
Abstract

$[NH_3(CH_2)_2NH_3][Mn(SO_4)_2(H_2O)_4]$ shows an alternate stacking of inorganic layers of tetraaquabis(sulfato-*O*)manganese anions, $[Mn(SO_4)_2(H_2O)_4]^{2-}$, and organic layers of $[NH_3(CH_2)_2NH_3]^{2+}$ cations. Anions and cations are linked together through N—H···O hydrogen bonds to form a three-dimensional network.

Comment

The X-ray structure determination of the title compound, (I), is a part of an investigation of the $MnSO_4$–$NH_2(CH_2)_2NH_2$–H_2SO_4 system. The manganese coordination polyhedron is a distorted octahedron built up from four water molecules and two sulfate anions. The Mn—O distances range from 2.160 (2) to 2.189 (2) Å.



(I)

The $[Mn(SO_4)_2(H_2O)_4]^{2-}$ octahedra are located on inversion centres. The sulfate anions are monodentate ligands. The main geometric features of the $[Mn(SO_4)_2(H_2O)_4]^{2-}$ anions are quite similar to those found for the inorganic $MnSO_4$ salt (Cox, Frazer & Will, 1965). The organic groups, $[NH_3(CH_2)_2NH_3]^{2+}$, are centrosymmetrical and present a *trans* configuration. The principal geometrical features of these groups are similar to those observed in some other organic sulfates, such as $NH_3(CH_2)_2NH_3SO_4$ (Benghozlen, Kamoun, Paulush & Pabst, 1994) and $NiNH_3(CH_2)_2NH_3(SO_4)_2.6H_2O$ (Healy, Patrick & White, 1984).

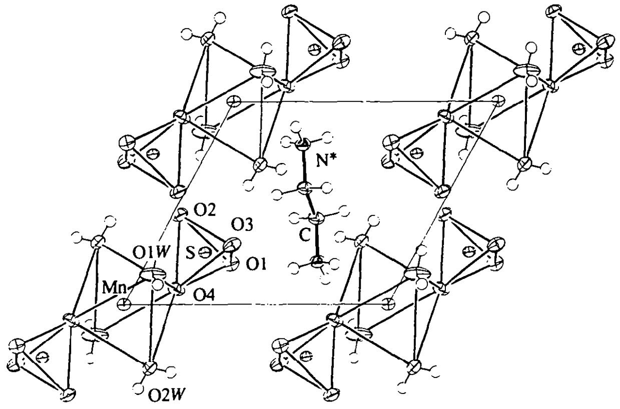


Fig. 1. *ORTEP*II (Johnson, 1976) projection onto the *bc* plane of $[NH_3(CH_2)_2NH_3][Mn(SO_4)_2(H_2O)_4]$. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii. * refers to symmetry code *x*, *y*, 1 + *z*.

Experimental

Crystals of $[NH_3(CH_2)_2NH_3][Mn(SO_4)_2(H_2O)_4]$ can easily be grown from aqueous or ethanolic solutions of manganese sulfate, ethylenediamine and sulfuric acid. They grow as flat rhombic platelets with the characteristic pink colouration of octahedral Mn^{II}. The density D_m was measured by picnometry.

Crystal data

$(C_2H_{10}N_2)[Mn(SO_4)_2(H_2O)_4]$
$M_r = 381.24$
Triclinic
$P\bar{1}$
$a = 6.850 (2)$ Å
$b = 7.188 (2)$ Å
$c = 7.304 (2)$ Å
$\alpha = 74.39 (2)^\circ$
$\beta = 72.02 (2)^\circ$
$\gamma = 78.79 (2)^\circ$
$V = 327.0 (2)$ Å^3
$Z = 1$
$D_x = 1.936$ Mg m^{-3}
$D_m = 1.951$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 23 reflections
$\theta = 14$–16°
$\mu = 1.391$ mm^{-1}
$T = 293 (2)$ K
Spherical
0.30 × 0.30 × 0.30 mm
Pink

Data collection

CAD-4 diffractometer
$\omega/2\theta$ scans [width $(1.05 + 0.35\tan\theta)^\circ$ in ω]
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.91$, $T_{\max} = 0.99$
1311 measured reflections
1145 independent reflections

1113 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0173$
$\theta_{\max} = 24.95^\circ$
$h = -7 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = 0 \rightarrow 8$
2 standard reflections
frequency: 60 min
intensity decay: 0.13%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0329$
$wR(F^2) = 0.1014$
$S = 1.167$
1145 reflections
125 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 0.2061P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.058$
$\Delta\rho_{max} = 0.786$ e Å^{-3}
$\Delta\rho_{min} = -0.426$ e Å^{-3}
Extinction correction: SHELXL93 (Sheldrick, 1993)
Extinction coefficient: 0.13 (1)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Mn1	0	0	0	0.0173 (3)
O1*W*	0.0862 (4)	−0.2491 (3)	0.2147 (3)	0.0380 (6)
O2*W*	0.2657 (3)	−0.0641 (3)	−0.2429 (3)	0.0285 (5)
S1	0.19778 (8)	0.22659 (8)	0.24955 (8)	0.0161 (3)
O1	0.3129 (3)	0.3974 (3)	0.1826 (3)	0.0241 (5)
O2	−0.0039 (3)	0.2683 (3)	0.3871 (3)	0.0293 (5)
O3	0.3172 (3)	0.0568 (3)	0.3436 (3)	0.0281 (5)
O4	0.1639 (3)	0.1906 (3)	0.0710 (2)	0.0260 (5)
N	0.3315 (4)	0.4954 (3)	−0.2427 (3)	0.0231 (5)
C	0.5491 (4)	0.5816 (4)	0.4185 (4)	0.0223 (6)

Table 2. *Selected geometric parameters (Å, °)*

Mn1—O1*W*^{i}	2.160 (2)	S1—O1	1.478 (2)
Mn1—O4^{i}	2.179 (2)	S1—O4	1.487 (2)
Mn1—O2*W*	2.189 (2)	N—C^{ii}	1.477 (3)
S1—O3	1.462 (2)	C—C^{iii}	1.519 (5)
S1—O2	1.467 (2)		
O1*W*^{i}—Mn1—O1*W*	180.0	O3—S1—O2	110.3 (1)
O1*W*—Mn1—O4^{i}	87.03 (8)	O3—S1—O1	110.3 (1)
O1*W*—Mn1—O4	92.97 (8)	O2—S1—O1	110.3 (1)
O4^{i}—Mn1—O4	180.0	O3—S1—O4	110.3 (1)
O1*W*—Mn1—O2*W*	94.25 (9)	O2—S1—O4	108.8 (1)
O4^{i}—Mn1—O2*W*	88.02 (8)	O1—S1—O4	106.8 (1)
O4—Mn1—O2*W*	91.98 (8)	S1—O4—Mn1	138.2 (1)
O1*W*—Mn1—O2*W*^{i}	85.75 (9)	N^{ii}—C—C^{iii}	109.5 (3)
O2*W*—Mn1—O2*W*^{i}	180.0		

Symmetry codes: (i) $-x, -y, -z$; (ii) $1 - x, 1 - y, -z$; (iii) $1 - x, 1 - y, 1 - z$.

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1O1*W*···O1^{i}	0.88 (4)	1.87 (4)	2.736 (3)	169 (3)
O1*W*—H2O1*W*···O2^{ii}	0.74 (5)	2.02 (5)	2.756 (3)	172 (5)
O2*W*—H1O2*W*···O3^{iii}	0.70 (4)	2.05 (4)	2.729 (3)	162 (4)
O2*W*—H2O2*W*···O3^{iv}	0.78 (6)	2.07 (6)	2.838 (3)	168 (5)
N—H1N···O4	0.86 (4)	1.98 (4)	2.832 (3)	172 (4)
N—H2N···O2^{v}	0.86 (4)	2.02 (4)	2.837 (3)	156 (3)
N—H3N···O1^{vi}	0.91 (4)	2.02 (4)	2.880 (3)	156 (3)

Symmetry codes: (i) $x, y - 1, z$; (ii) $-x, -y, 1 - z$; (iii) $1 - x, -y, -z$; (iv) $x, y, z - 1$; (v) $-x, 1 - y, -z$; (vi) $1 - x, 1 - y, -z$.

Data were corrected for Lorentz–polarization and absorption effects. The structure was solved by Patterson methods. Refinement was by full-matrix least-squares methods.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1123). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetrakis(triethylammonium) Octamolybdate(VI) Dihydrate

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Abstract

The X-ray structure analysis of the title compound, $(C_6H_{16}N)_4[Mo_8O_{26}].2H_2O$, has confirmed the presence of the $[Mo_8O_{26}]^{4-}$ anion. Eight edge- and corner-sharing MoO_6 octahedra constitute the anion, which has short terminal Mo—O bonds [1.684 (3)–1.711 (3) Å], bonds of intermediate length [1.749 (3)–1.990 (3) Å] and long bonds [2.140 (2)–2.472 (3) Å]. The structure consists of β-Mo_8O_{26} polyanions, triethylammonium

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