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Hexakis(dimethyl sulfoxide)manganese(II) bis(perchlorate)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(I–O) = 0.009 Å
Disorder in main residue
R factor = 0.044
wR factor = 0.116
Data-to-parameter ratio = 17.5

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

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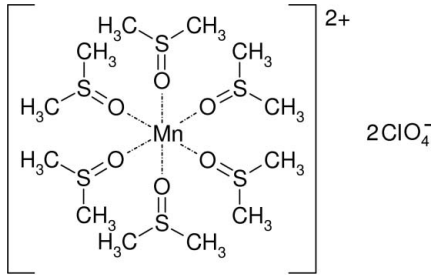
The title compound, $[Mn(C_2H_6OS)_6](ClO_4)_2$, comprises discrete $[Mn(DMSO)_6]^{2+}$ cations (DMSO is dimethyl sulfoxide) and perchlorate anions, all of which lie on crystallographic twofold axes. The O atoms of six DMSO molecules are bonded to the Mn^{II} ion in a distorted octahedral geometry.

Comment

Previously, we have characterized the title compound, (I), by the differential scanning calorimetry technique (Migdał-Mikuli & Szostak, 2005). We now report here the crystal structure of the title compound, (I).



(I)

The structure of (I) comprises discrete $[Mn(DMSO)_6]^{2+}$ [DMSO = $(CH_3)_2SO$, dimethyl sulfoxide] cations and perchlorate anions, all of which lie on crystallographic twofold axes (Fig. 1). The O atoms of six DMSO molecules are bonded

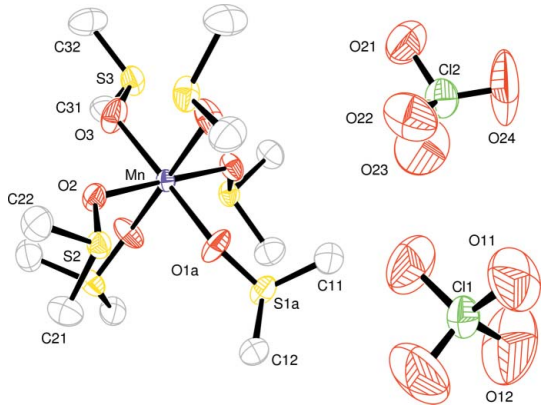


Figure 1
The structure unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Unlabelled atoms in the $[Mn(DMSO)_6]^{2+}$ cation and one of the anions are related to labelled atoms by the symmetry operations $(-x, -y, z)$ and $(\frac{1}{2} - x, \frac{3}{2} - y, z)$, respectively. The minor component of a DMSO ligand, one component of one perchlorate anion, and H atoms have been omitted for clarity.

to the Mn^{II} ion in a distorted octahedral geometry (Table 1). Compound (I) is isostructural with its cadmium(II) analogue reported earlier (Sandström, 1978; Lubeznowa & Ponomariew, 1989).

Experimental

Hexaaquamanganese(II) perchlorate was synthesized by the reaction of manganese carbonate (13.8 mmol) with diluted $HClO_4$ (27.6 mmol). The hexaaquamanganese(II) perchlorate (13.8 mmol) was dissolved while being slowly heated in dimethyl sulfoxide (DMSO, 83.0 mmol); the solution was then cooled to obtain a precipitate of compound (I) (13.8 mmol). It was filtered and then dried in a desiccator over phosphorus pentoxide for a few hours. Single crystals suitable for X-diffraction study were obtained by repeated recrystallization from DMSO solution.

Crystal data

$[Mn(C_2H_6OS)_6](ClO_4)_2$
M_r = 722.61
Orthorhombic, *Fdd*2
a = 25.4415 (6) Å
b = 12.4043 (3) Å
c = 20.1696 (5) Å
V = 6365.2 (3) Å^3
Z = 8
D_x = 1.508 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.03 mm^{-1}
T = 293 (2) K
Prism, colourless
0.18 × 0.18 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.837, T_{max} = 0.904
3664 measured reflections
3532 independent reflections
2777 reflections with $I > 2\sigma(I)$
R_{int} = 0.06
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.116
S = 1.03
3532 reflections
202 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 7.9989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00036 (8)
Absolute structure: Flack (1983), 1655 Friedel pairs
Flack parameter: 0.00 (17)

Table 1
Selected geometric parameters (Å, °).

Mn—O1*B*	2.144 (18)	Mn—O3	2.169 (3)
Mn—O1*A*	2.157 (10)	Mn—O2	2.181 (3)
O1*B*—Mn—O1*A*^{i}	84.0 (5)	O3—Mn—O2^{i}	91.21 (13)
O1*A*—Mn—O1*A*^{i}	99.7 (6)	O1*A*—Mn—O2	85.7 (3)
O1*A*—Mn—O3	174.3 (3)	O3—Mn—O2	89.57 (12)
O1*A*—Mn—O3^{i}	83.6 (3)	O2^{i}—Mn—O2	178.86 (18)
O3—Mn—O3^{i}	93.5 (3)		

Symmetry code: (i) $-x, -y, z$.

The crystal was identified as being twinned. The twin law was found to be (100 010 00$\bar{1}$), and the major twin fraction refined to 0.58 (3). The S and O atoms (O1 and S1) of one of the independent dimethyl sulfoxide groups are disordered over two positions with occupation factors of 0.639 (4) and 0.361 (4). One of the perchlorate anions (with Cl2) is disordered about a twofold rotation axis and its O atoms were refined with an occupation factor of 0.50. The Cl—O lengths and O···O distances were restrained to 1.40 (3) and 2.29 (1) Å, respectively. The methyl H atoms were positioned geometrically (C—H = 0.96 Å) and refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(C).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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