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Poly[[aquatris(μ-dimethyl phosphato-κ^2O,O')-bis(μ-dimethyl sulfoxide-κO)(dimethyl sulfoxide-κO)-dimanganese(II)] nitrate monohydrate]

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In the title compound, $\{[Mn_2\{(O_2P(OCH_3)_2\}_3(C_2H_6SO)_3(H_2O)]NO_3 \cdot H_2O\}_n$, the octahedral Mn^{II} ions form a polymeric chain, being bridged by both dimethyl sulfoxide molecules and dimethyl phosphate anions. A network of O—H···O hydrogen bonds helps to consolidate the crystal packing.

Comment

In a recent paper, we reported the synthesis and crystal structure of $[Ni(H_2O)_6](DMP)_2$ (Rafizadeh & Amani, 2006a), where DMP is the dimethyl phosphate anion, $[O_2P(OCH_3)_2]^-$. In this compound, DMP is not bonded to the metal and acts as a counter-ion. Conversely, in $[Cu_2(\mu\text{-DMP})_4(DMSO)]_n$ (Rafizadeh et al., 2005), $[UO_2(\mu\text{-DMP})_4(DMSO)]_n$ (Rafizadeh, Hoseinzadeh & Amani, 2006), $[La(\mu\text{-DMP})_2(\mu_3\text{-}NO_3)(DMSO)]_n$ (Rafizadeh, Amani & Broushaky, 2006) and $[UO_2(\mu\text{-DEP})_4(DMSO)]_n$ (Rafizadeh & Amani, 2006b), (DMSO is dimethyl sulfoxide and DEP is diethyl phosphate), DMP and DEP act as O-atom donor ligands, thus forming coordination polymers in the solid state. We now report the synthesis and structure of the polymeric title compound, (I).

(I)
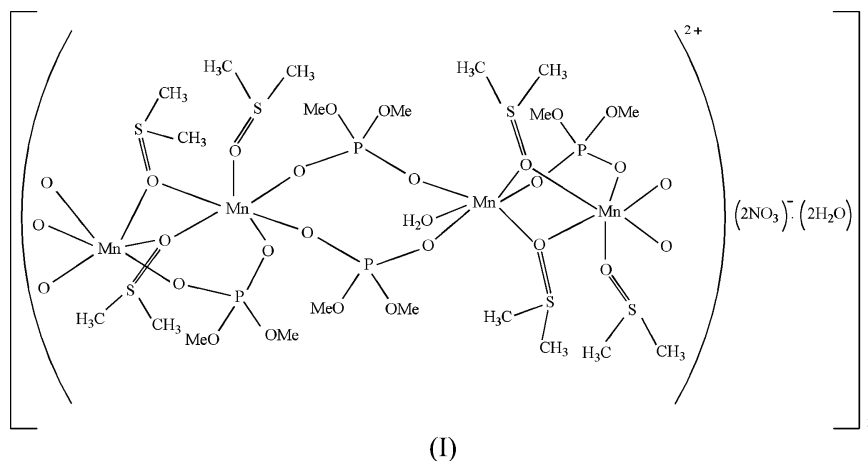
The asymmetric unit of (I) contains four distinct Mn centers that adopt distorted MnO_6 octahedral coordination. In each case, five of the attached O atoms arise from bridging DMP anions and DMSO molecules. The sixth coordination site is occupied by a non-bridging DMSO O atom (Mn1 and Mn3) or a water molecule O atom (Mn2 and Mn4). Overall, polymeric chains propagating in [100] result. There are also two NO_3^- counter-ions and two non-coordinated water molecules in the asymmetric unit (Fig. 1). The Mn—O (Table 1) and P—O bond lengths in (I) are in agreement with the corresponding
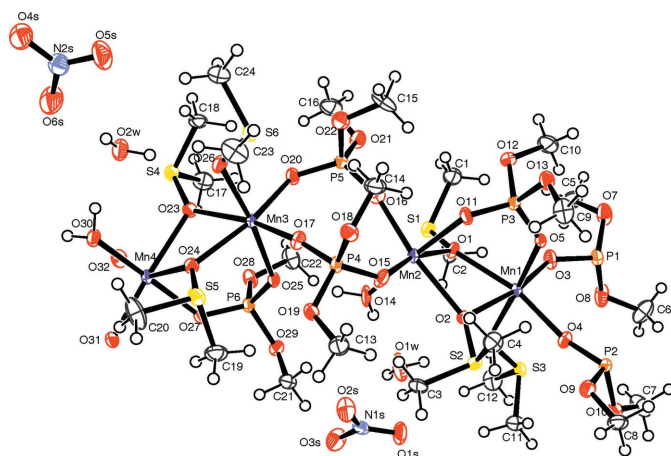
Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level (arbitrary spheres for the H atoms).
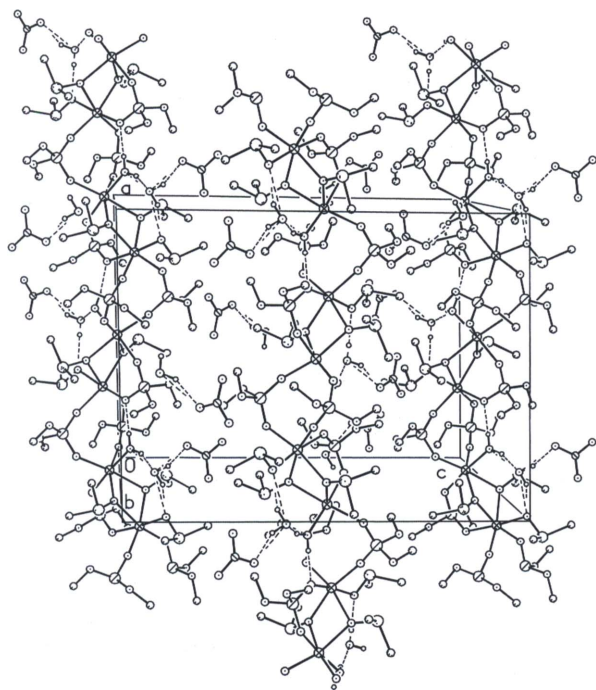

Figure 2
Packing diagram for (I). Hydrogen bonds are shown as dashed lines.

ones in [Mn(HPO$_4$)(H$_2$O)$_3$] (Krishnamohan Sharma *et al.*, 2003).

An extensive network of O—H···O hydrogen bonds (Table 2) helps to consolidate the crystal packing (Fig. 2).

Experimental

Trimethyl phosphate (2.17 g, 1.8 ml, 15 mmol) was added to a solution of Mn(NO$_3$)$_2$·4H$_2$O (0.97 g, 3.75 mmol) in DMSO (10 ml) and ethanol (20 ml) and the resulting colorless solution was refluxed at 338 K for 3 h. This solution was left to evaporate slowly at room temperature. After six months, colorless prismatic crystals of (I) were isolated (yield 1.12 g, 73.1%; m.p. 342 K).

Crystal data

[Mn$_2$(C$_2$H$_6$O$_4$P)$_3$(C$_2$H$_6$SO)$_3$-(H$_2$O)]·NO$_3$·H$_2$O	$V = 6456.1$ (16) Å^3
$M_r = 817.42$	$Z = 8$
Monoclinic, $P2_1/c$	$D_x = 1.682$ Mg m^{-3}
$a = 15.398$ (2) Å	Mo $K\alpha$ radiation
$b = 20.877$ (3) Å	$\mu = 1.20$ mm^{-1}
$c = 20.090$ (3) Å	$T = 120$ (2) K
$\beta = 91.561$ (4)°	Prism, colorless
	$0.20 \times 0.15 \times 0.15$ mm

Data collection

Bruker SMART1000 CCD diffractometer	71520 measured reflections
ω scans	14050 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)	7711 reflections with $I > 2\sigma(I)$
$T_{min} = 0.754$, $T_{max} = 0.835$	$R_{int} = 0.094$
	$\theta_{max} = 27.0°$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.0185P)^2]$
$wR(F^2) = 0.090$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.002$
14050 reflections	$\Delta\rho_{max} = 1.20$ e Å^{-3}
763 parameters	$\Delta\rho_{min} = -0.55$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Mn1—O4	2.100 (3)	Mn3—O17	2.098 (3)
Mn1—O3	2.118 (3)	Mn3—O20	2.112 (3)
Mn1—O5	2.152 (3)	Mn3—O25	2.163 (3)
Mn1—O1	2.237 (3)	Mn3—O26	2.229 (3)
Mn1—O6	2.241 (3)	Mn3—O23	2.241 (3)
Mn1—O2	2.254 (3)	Mn3—O24	2.252 (3)
Mn2—O16	2.083 (3)	Mn4—O32	2.094 (3)
Mn2—O11	2.098 (3)	Mn4—O27	2.122 (3)
Mn2—O14	2.128 (3)	Mn4—O31	2.132 (3)
Mn2—O15	2.126 (3)	Mn4—O30	2.143 (3)
Mn2—O2	2.309 (3)	Mn4—O24	2.282 (3)
Mn2—O1	2.319 (3)	Mn4—O23	2.320 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O14—H14D···O25	0.85	1.90	2.752 (4)	178
O14—H14E···O1W	0.85	1.90	2.698 (5)	156
O30—H30D···O2W	0.85	1.93	2.724 (4)	156
O30—H30E···O5^i	0.85	1.92	2.761 (4)	168
O1W—H1W1···O2S	0.85	1.98	2.766 (5)	153
O1W—H1W2···O6	0.85	2.06	2.845 (4)	154
O2W—H2W1···O6S	0.85	1.99	2.770 (5)	152
O2W—H2W2···O26	0.85	1.99	2.837 (4)	177

Symmetry code: (i) $x - 1, y, z$.

The O-bound H atoms were located in a difference map and refined as riding in their as-found relative positions, with U_{iso}(H) = 1.5U_{eq}(O). The C-bound H atoms were placed in idealized locations (C—H = 0.98 Å) and refined as riding, with U_{iso}(H) = 1.5U_{eq}(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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