Crystal structure of bis(diaquamanganese(II))hexaacetatomanganese(II) octahydrate, $((\text{H}_2\text{O})_2\text{Mn})_2(\text{C}_2\text{H}_3\text{O}_2)_6\text{Mn} \cdot 8\text{H}_2\text{O}$

S.-Y. Li^I, Y.-H. Li^I, H.-L. Liu^I, H.-L. Zhu^{*,I} and D.-Q. Wang^{II}^I Wuhan Institute of Science and Technology, Department of Environmental and Chemical Engineering, Wuhan, 430073 P. R. China^{II} Liaocheng University, Department of Chemistry, Liaocheng, 252059 P. R. China

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Abstract

$\text{C}_{12}\text{H}_{42}\text{Mn}_3\text{O}_{24}$, monoclinic, $P12_1/c1$ (No. 14), $a = 10.409(8)$ Å, $b = 17.52(1)$ Å, $c = 9.059(7)$ Å, $\beta = 110.93(1)^\circ$, $V = 1543.3$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.032$, $wR_{\text{ref}}(F^2) = 0.089$, $T = 298$ K.

Source of material

Commercial $\text{Mn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (OAc = acetate anion) was dissolved to anhydrous methanol solution, to which diethyl ether was diffused for three days. Large pink plate-like crystals were precipitated, filtered and washed with methanol for three times, and dried in a vacuum under CaCl_2 . Elemental analysis: found – C, 19.81%; H, 5.88%; calc. for $\text{C}_4\text{H}_{14}\text{MnO}_8$ – C, 19.60%; H, 5.76%.

Experimental details

Because the H atoms attached to the water molecules were not able to be calculated theoretically, these hydrogen atoms were located from difference Fourier maps and refined isotropically.

Discussion

The title complex is a three-dimensional polymeric one. There are two kinds of univalent Mn(II) atoms in each trinuclear Mn unit. Mn(1) atom in the complex is six-coordinated to six oxygen atoms from four different acetate anions and two water molecules, forming a distorted octahedron (the diagonal angles are $168.79(7)^\circ$, $170.22(7)^\circ$ and $177.09(6)^\circ$). Mn(2) is surrounded by six oxygen atoms from six different acetate anions also to constitute a distorted octahedron (the diagonal angles are $180.00(9)^\circ$, $180.0(1)^\circ$ and $180.00(3)^\circ$). Mn(1) and Mn(2) atoms are bridged

together by three different acetate anion ligands, two of which adopt dinucleated O, O' -bridged coordination mode to ligate to the two metal atoms, and the third acetate ligand is in O, O, O' -bridged mode to three Mn atoms. Each of the donor oxygen atoms in the acetates with O, O, O' -bridged mode coordinates to another trimer manganese unit, which connects the complex to form a two-dimensional network along bc -plane. Uncoordinated water molecules join the neighboring planes by hydrogen bonds to form a three-dimensional network, in which small tunnels with the diameter of about 4 Å are regularly distributed.

Table 1. Data collection and handling.

Crystal:	pink plate, size $0.20 \times 0.33 \times 0.52$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	12.97 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{\text{max}}$:	52.76°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	8657, 3098
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2423
$N(\text{param})_{\text{refined}}$:	226
Programs:	SHELXTL [1], SHELXTL-plus [2]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2A)	4e	−0.3544	0.0274	0.1649	0.088
H(2B)	4e	−0.3891	0.1145	0.1376	0.088
H(2C)	4e	−0.2918	0.0855	0.3036	0.088
H(4A)	4e	0.3649	0.1086	0.5169	0.095
H(4B)	4e	0.4136	0.0447	0.4275	0.095
H(4C)	4e	0.3119	0.0247	0.5144	0.095
H(6A)	4e	−0.0049	0.0363	−0.3229	0.073
H(6B)	4e	0.0843	0.0914	−0.3834	0.073
H(6C)	4e	−0.0762	0.0995	−0.4477	0.073
H(1)	4e	−0.162(3)	0.282(1)	−0.174(2)	0.050(9)
H(2)	4e	−0.228(3)	0.270(2)	−0.064(4)	0.08(1)
H(3)	4e	0.193(3)	0.285(1)	−0.026(2)	0.053(9)
H(4)	4e	0.274(3)	0.275(2)	0.133(3)	0.08(1)
H(5)	4e	0.725(3)	0.157(2)	0.808(5)	0.12(2)
H(6)	4e	0.701(4)	0.077(1)	0.783(6)	0.12(2)
H(7)	4e	0.411(6)	0.360(2)	0.339(6)	0.15(2)
H(8)	4e	0.478(5)	0.288(3)	0.380(5)	0.19(3)
H(9)	4e	0.546(4)	0.270(2)	0.929(6)	0.13(2)
H(10)	4e	0.602(9)	0.326(5)	1.046(9)	0.43(7)
H(11)	4e	0.706(5)	0.903(3)	1.097(2)	0.15(3)
H(12)	4e	0.729(5)	0.899(4)	0.954(6)	0.23(4)

* Correspondence author (e-mail: hlzhu@wist.edu.cn)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Mn(1)	4e	0.02019(4)	0.20172(2)	0.08744(4)	0.0335(2)	0.0203(2)	0.0263(2)	0.0006(1)	0.0105(2)	−0.0029(1)
Mn(2)	2a	0	0	0	0.0283(3)	0.0170(2)	0.0270(3)	−0.0001(2)	0.0092(2)	0.0006(2)
O(1)	4e	−0.1370(2)	0.15178(9)	0.1677(2)	0.047(1)	0.0309(9)	0.045(1)	−0.0078(8)	0.0248(9)	−0.0056(8)
O(2)	4e	−0.1638(2)	0.03336(9)	0.0780(2)	0.042(1)	0.0310(9)	0.051(1)	−0.0009(8)	0.0263(9)	−0.0058(8)
O(3)	4e	0.1744(2)	0.14712(9)	0.2862(2)	0.047(1)	0.0288(9)	0.037(1)	0.0075(8)	0.0062(8)	0.0015(8)
O(4)	4e	0.1714(2)	0.02628(9)	0.2129(2)	0.041(1)	0.0322(9)	0.034(1)	−0.0010(8)	0.0018(8)	−0.0044(8)
O(5)	4e	0.0102(2)	0.11559(8)	−0.0951(2)	0.038(1)	0.0193(7)	0.0251(8)	−0.0004(7)	0.0131(7)	0.0006(6)
O(6)	4e	0.0244(2)	0.21093(8)	−0.2453(2)	0.052(1)	0.0215(8)	0.032(1)	−0.0009(7)	0.0169(8)	0.0050(7)
O(7)	4e	−0.1621(2)	0.2537(1)	−0.0946(2)	0.038(1)	0.038(1)	0.035(1)	0.0078(8)	0.0163(8)	0.0075(8)
O(8)	4e	0.2047(2)	0.2548(1)	0.0561(2)	0.043(1)	0.046(1)	0.033(1)	−0.0107(9)	0.0117(9)	0.0002(9)
O(9)	4e	0.6676(2)	0.1221(1)	0.7560(3)	0.048(1)	0.048(1)	0.054(1)	0.002(1)	0.008(1)	0.003(1)
O(10)	4e	0.4280(2)	0.3172(2)	0.3021(3)	0.054(1)	0.066(2)	0.050(1)	−0.005(1)	0.014(1)	−0.005(1)
O(11)	4e	0.6162(3)	0.3033(2)	0.9655(4)	0.066(2)	0.095(2)	0.085(2)	0.001(2)	0.037(2)	−0.016(2)
O(12)	4e	0.6658(2)	0.8951(1)	0.9956(4)	0.049(1)	0.063(2)	0.085(2)	−0.004(1)	0.033(1)	0.003(1)
C(1)	4e	−0.1981(3)	0.0890(1)	0.1430(3)	0.033(1)	0.026(1)	0.029(1)	0.003(1)	0.011(1)	0.002(1)
C(2)	4e	−0.3191(3)	0.0781(2)	0.1916(4)	0.051(2)	0.057(2)	0.083(2)	−0.010(2)	0.041(2)	−0.019(2)
C(3)	4e	0.2186(2)	0.0802(1)	0.3070(3)	0.029(1)	0.029(1)	0.032(1)	−0.001(1)	0.007(1)	0.000(1)
C(4)	4e	0.3379(4)	0.0630(2)	0.4546(4)	0.062(2)	0.042(2)	0.054(2)	0.012(2)	−0.017(2)	−0.006(2)
C(5)	4e	0.0133(2)	0.1419(1)	−0.2262(3)	0.029(1)	0.023(1)	0.027(1)	0.0015(9)	0.009(1)	0.001(1)
C(6)	4e	0.0032(4)	0.0874(1)	−0.3567(3)	0.091(2)	0.029(1)	0.033(2)	−0.003(1)	0.030(2)	−0.003(1)

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