factors of all non-H atoms are given in Table 1.* Selected interatomic distances and angles for both enantiomers are listed in Table 2.

Related literature. Other structures containing tris-pyrazolylborate groups coordinated to rhodium have been reported, including $[Rh^{III}(Bpz_4)(CO)(I)_2]$ (Cocivera, Desmond, Ferguson, Kaitner, Lalor & O'Sullivan, 1982), $[Rh^{II}(C_5Me_5)(HBpz_3)][PF_6]$ (Restivo, Ferguson, O'Sullivan & Lalor, 1975), and $[Rh^{III}\{HB(3,5\text{-}Me_2pz)_3\}(C_2H_5)(C_6H_5)(CO)]$ (3,5-$Me_2pz = 3,5$-dimethylpyrazolyl) (Ghosh & Graham, 1989).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54020 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

This structure was solved at the X-ray Crystallographic Facility (CHEXRAY) at the Department of Chemistry, University of California, Berkeley, as part of the requirements of a graduate chemistry course on structure analysis by X-ray diffraction. We thank Mr Liang Tong, Dr F. Hollander and Professor D. H. Templeton for their guidance.

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Structure of Manganese Acetate Dihydrate

By Chih-Yi Cheng and Sue-Lein Wang*

Department of Chemistry, National Tsing Hua University, Hsinchu, Taiwan 30043

Abstract. Hexaacetatotetraaquatrimanganese dihydrate, $[Mn_3(CH_3COO)_6(H_2O)_4].2H_2O$, $M_r = 627.3$, monoclinic, $P2_1/c$, $a = 8.982$ (4), $b = 9.155$ (4), $c = 16.514$ (5) Å, $\beta = 114.18$ (3)°, $V = 1239$ (1) Å^3, $Z = 2$, $D_x = 1.678$ Mg m^{-3}, $\lambda = 0.71073$ Å, $\mu = 1.518$ mm^{-1}, $F(000) = 642$, $T = 297$ K, $R = 0.044$, $wR = 0.044$ for 1271 unique reflections with $I > 3.0\sigma(I)$. The title compound consists of MnO$_6$ octahedra which are linked by acetate bridges to form Mn$_3$(OAc)$_6$(H$_2$O)$_4$ units in the bc plane. This structure is closely related to that of the octahydrate Mn$_3$(CH$_3$COO)$_6$(H$_2$O)$_4$.8H$_2$O [Bertaut, Duc, Burlet, Burlet, Thomas & Moreau (1974). *Acta Cryst.* B30, 2234–2236]. In the crystal structure of the title compound, two thirds of the water molecules are in the manganese coordination sphere and the rest reside in the interlayer region. Two adjacent layers are 8.2 Å apart and held by hydrogen bonds through water and acetate moieties.

Experimental. The sample was obtained as pink crystals by heating a reaction mixture of Mn$_3$O$_4$

(0.6g) and 14.0 ml 12.5 *M* acetic acid in a 23 ml teflon-lined autoclave at 503 K for 2 d followed by slow cooling to room temperature. The structure of Mn$_3$(CH$_3$COO)$_6$(H$_2$O)$_4$.2H$_2$O was determined from single-crystal X-ray diffraction. Peak profile analysis (ω scan) on the pink plate crystals using a Nicolet $R3m/V$ diffractometer with Mo $K\alpha$ radiation indicated that most of them were not suitable for indexing and intensity data collection. Many had to be selected from the reaction products before a satisfactory crystal was obtained. Finally a piece of dimensions 0.06 × 0.10 × 0.12 mm was chosen. Some of its reflections also showed broad peak profiles but the quality of the intensity data was considered to be adequate for elucidation of the structure. The unit-cell parameters were determined by a least-squares fit of 15 reflections with 2θ ranging from 12.8 to 27.9°. The intensity data were collected up to $2\theta = 50°$ ($\sin\theta_{max}/\lambda = 0.594$ Å^{-1}) with $\theta/2\theta$ scans. A periodic check of three standard reflections did not reveal any significant variation in intensity. A total of 3587 reflections were measured in the range $-10 \le h \le 10$, $0 \le k \le 10$, $0 \le l \le 19$. Scan width 1.20° plus $K\alpha$ separation and scan speed 2.93–14.95° min^{-1}. The

* To whom correspondence should be addressed.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($\text{Å}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Mn(1)	0	5000	5000	17 (1)
Mn(2)	−49 (1)	721 (1)	7978 (1)	22 (1)
O(1)	1516 (5)	6146 (5)	6236 (3)	24 (2)
O(2)	3412 (6)	7569 (6)	7185 (3)	40 (2)
O(3)	−632 (6)	2440 (5)	7021 (3)	30 (2)
O(4)	−721 (6)	3971 (5)	5984 (3)	23 (2)
O(5)	−1887 (6)	6632 (5)	4756 (3)	28 (2)
O(6)	−1770 (6)	7202 (5)	6087 (3)	32 (2)
O(7)	1781 (6)	95 (6)	7484 (3)	39 (2)
O(8)	−2187 (5)	−507 (5)	7085 (3)	29 (2)
O(W)	3249 (6)	2567 (6)	7006 (3)	36 (2)
C(1)	2958 (8)	6717 (8)	6557 (4)	27 (3)
C(2)	4096 (9)	6290 (9)	6140 (5)	40 (3)
C(3)	−783 (8)	2656 (7)	6252 (4)	20 (2)
C(4)	−1053 (11)	1437 (8)	5618 (5)	41 (3)
C(5)	−2408 (8)	7286 (7)	5256 (4)	23 (3)
C(6)	−3902 (10)	8217 (9)	4818 (5)	44 (3)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Mn(1)—O(1)	2·201 (4)	Mn(1)—O(4)	2·194 (5)
Mn(1)—O(5)	2·170 (5)	Mn(1)—O(1a)	2·201 (4)
Mn(1)—O(4a)	2·194 (5)	Mn(1)—O(5a)	2·170 (5)
Mn(2)—O(3)	2·138 (5)	Mn(2)—O(7)	2·188 (7)
Mn(2)—O(8)	2·192 (4)	Mn(2)—O(1b)	2·231 (6)
Mn(2)—O(4b)	2·239 (4)	Mn(2)—O(6b)	2·197 (4)
O(1)—C(1)	1·292 (8)	O(2)—C(1)	1·225 (9)
O(3)—C(3)	1·237 (9)	O(4)—C(3)	1·291 (8)
O(5)—C(5)	1·255 (10)	O(6)—C(5)	1·254 (8)
C(1)—C(2)	1·499 (13)	C(3)—C(4)	1·481 (10)
C(5)—C(6)	1·501 (10)	O(7)—O(W)	2·887 (8)
O(W)—O(2b)	2·739 (7)	O(W)—O(5a)	2·754 (6)
O(W)—O(8c)	2·728 (8)	O(8)—O(6d)	2·787 (7)

O(1)—Mn(1)—O(4)	77·1 (1)	O(1)—Mn(1)—O(5)	89·1 (2)
O(4)—Mn(1)—O(5)	89·3 (2)	O(1)—Mn(1)—O(1a)	180·0 (1)
O(4)—Mn(1)—O(1a)	102·9 (2)	O(5)—Mn(1)—O(1a)	90·9 (2)
O(1)—Mn(1)—O(4a)	102·9 (2)	O(4)—Mn(1)—O(4a)	180·0 (1)
O(5)—Mn(1)—O(4a)	90·7 (2)	O(1)—Mn(1)—O(5a)	90·9 (2)
O(4)—Mn(1)—O(5a)	90·7 (2)	O(5)—Mn(1)—O(5a)	180·0 (1)
O(3)—Mn(2)—O(7)	84·9 (2)	O(3)—Mn(2)—O(8)	89·9 (2)
O(7)—Mn(2)—O(8)	101·5 (2)	O(3)—Mn(2)—O(1b)	106·1 (2)
O(7)—Mn(2)—O(1b)	167·2 (2)	O(8)—Mn(2)—O(1b)	85·2 (2)
O(3)—Mn(2)—O(4b)	176·5 (2)	O(7)—Mn(2)—O(4b)	93·1 (2)
O(8)—Mn(2)—O(4b)	93·4 (2)	O(1b)—Mn(2)—O(4b)	75·5 (2)
O(3)—Mn(2)—O(6b)	87·3 (2)	O(7)—Mn(2)—O(6b)	88·2 (2)
O(8)—Mn(2)—O(6b)	169·6 (2)	O(1b)—Mn(2)—O(6b)	85·9 (2)
O(4b)—Mn(2)—O(6b)	89·7 (2)	O(1)—C(1)—O(2)	122·3 (8)
O(1)—C(1)—C(2)	117·6 (6)	O(2)—C(1)—C(2)	120·1 (6)
O(3)—C(3)—O(4)	119·9 (6)	O(3)—C(3)—C(4)	121·5 (6)
O(4)—C(3)—C(4)	118·6 (6)	O(5)—C(5)—O(6)	124·3 (6)
O(5)—C(5)—C(6)	117·1 (6)	O(6)—C(5)—C(6)	118·7 (7)

Symmetry codes: (a) $-x$, $1 - y$, $1 - z$; (b) $-x$, $y - \frac{1}{2}$, $\frac{3}{2} - z$; (c) $-x$, $\frac{1}{2} + y$, $\frac{3}{2} - z$; (d) x, $y - 1$, z.

space group $P2_1/c$ (No. 14) was determined from the systematic absences $0k0$, $k = 2n + 1$; $h0l$, $l = 2n + 1$. No absorption correction was made because the difference between the values of T_{min} and T_{max} was very small. After corrections for Lorentz and polarization effects, 1271 unique structure amplitudes (R_{int} = 0·038) with $I > 3\sigma(I)$ were obtained and used in the structure refinement. The structure was solved by

direct methods and refined by full-matrix least squares based on F values. All the non-H atoms were refined with anisotropic temperature factors. For 1271 reflections and 151 variables, the refinement converged at $R = 0·044$, $wR = 0·044$, $w = [\sigma^2(F) + 0·0006F^2]^{-1}$, $\sigma^2(F)$ based on counting statistics, $(\Delta/\sigma)_{max} = 0·001$, $S = 1·14$, $(\Delta\rho)_{max} = 0·79$, $(\Delta\rho)_{min} = -0·41$ e Å^{-3}. The coordinates of the H atoms were calculated and refined with a fixed U (0·08 Å^2). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a DEC MicroVAX II computer system using *SHELXTL-Plus* (Sheldrick, 1990). Atomic parameters are given in Table 1,
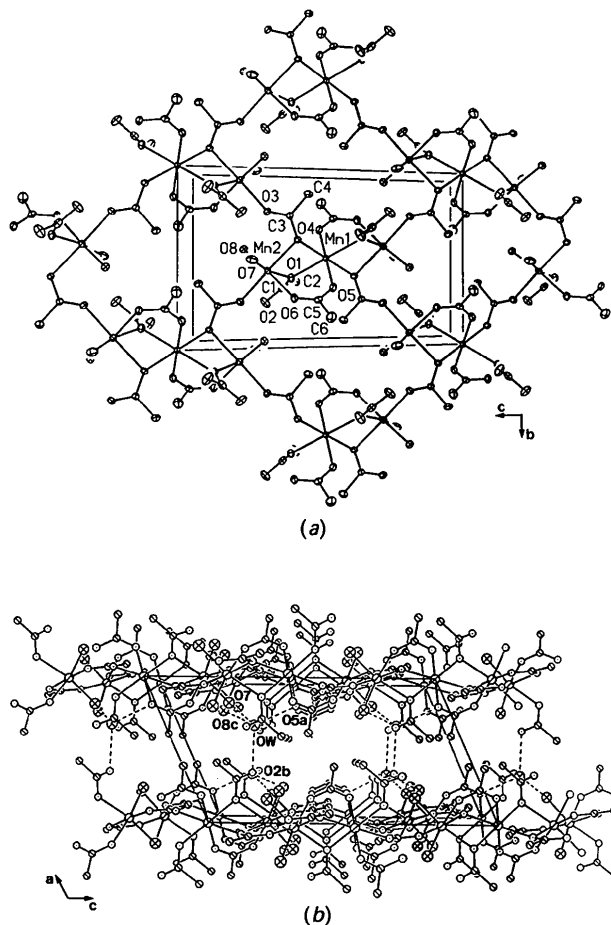

Fig. 1. *ORTEP* drawings of $Mn_3(CH_3COO)_6(H_2O)_4 \cdot 2H_2O$. (a) Projection in the bc plane showing the coordination environment of the Mn^{II} ions, (b) projection in the ac plane showing the sites of water molecules in the interlayer region. The Mn, O(OAc⁻), O (water in both the Mn coordination sphere and the interlayer region) and C atoms are represented by shaded bottom left to top right, dotted, cross-hatched and shaded bottom right to top left circles, respectively. The hydrogen bonds between water molecules and acetates are represented by dashed lines.

selected bond distances and angles in Table 2.* Projections of the structure in both the bc and the ac planes are shown in Fig. 1.

Related literature. The crystal structure of $Mn_3(CH_3COO)_6(H_2O)_4.2H_2O$ is closely related to that of the octahydrate $Mn_3(CH_3COO)_6(H_2O)_4.8H_2O$ which was previously reported as $Mn(CH_3COO)_2.4H_2O$ by Bertaut, Duc, Burlet, Burlet, Thomas & Moreau (1974). The layers comprise $Mn_3(OAc)_6(H_2O)_4$ units in both structures. In manganese acetate octahydrate, the two O atoms in all of the acetate groups

coordinate to two different Mn^{II} ions, whereas in the dihydrate only two thirds of the acetate groups are bridged in this way. The rest of the acetates coordinate to two Mn^{II} ions through only one O atom, O(1). In addition, the interlayer distance is larger (9·7 versus 8·2 Å) in $Mn_3(CH_3COO)_6(H_2O)_4.8H_2O$ because there are twelve more water molecules per unit cell in the interlayer region than in the structure of the dihydrate.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54005 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,7-Bis(benzimidazol-2-yl)-2,6-dithiaheptanecopper(I) Perchlorate at 233 K

BY R. CARBALLO AND A. CASTIÑEIRAS*

Departamento de Química Inorgánica, Facultad de Farmacia, Universidad de Santiago de Compostela, E-15706 Santiago de Compostela, Spain

AND W. HILLER AND J. STRÄHLE

Institut für Anorganische Chemie der Universität Tübingen, Auf der Morgenstelle 18, D-7400 Tübingen, Germany

Abstract. $[Cu(C_{19}H_{20}N_4S_2)]ClO_4$, $M_r = 531.52$, monoclinic, $C2/c$, $a = 14.166$ (4), $b = 17.571$ (5), $c = 9.942$ (2) Å, $\beta = 118.08$ (2)°, $V = 2183.3$ Å^3, $Z = 4$, $D_x = 1.617$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.54056$ Å, $\mu = 46.1$ cm^{-1}, $F(000) = 1088$, $T = 233$ K, the final R value is 0.053 for 1039 significant $[I > 3\sigma(I)]$ reflections. The Cu^I ion is only linearly coordinated by two pyridine N atoms of the benzimidazole groups of the BBDHP ligand [Cu—N = 1.908 (4) Å]. The thioether S atoms do not have significant bonding contacts with the copper ion [Cu—S = 2.876 (2) Å]. The cation and perchlorate anion are linked by a hydrogen bond.

Experimental. A colourless single crystal of approximate dimensions $0.10 \times 0.15 \times 0.10$ mm was sealed in a capillary. Because the crystals decompose at

room temperature all X-ray investigations were performed at 233 K. The systematic absences observed suggested the space group to be either Cc or $C2/c$. For calculations the centrosymmetric space group $C2/c$ was chosen, and later confirmed by successful refinement of the structure. In the final full-matrix least-squares refinement all non-H atoms were assigned anisotropic temperature parameters; each atom of the perchlorate anion was allowed an individual isotropic temperature factor. The disordered perchlorate anion was assumed to occupy two alternative orientations related by a crystallographic twofold axis, and their multiplicities were also refined, resulting in an occupancy of 0.5. The calculated H-atom positions were used only for structure factor calculations with B_{iso} fixed at 4 Å^2. More details of the intensity data collection, structure solution and refinement are listed in Table 1. Final atomic coordinates are given in Table 2, selected distances and

* To whom correspondence should be addressed.