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[2-(*N*,*N*-Dimethylamino)ethanol-$\kappa^2 N,O$]-bis(2,4-pentanedionato-$\kappa^2 O,O'$)manganese(II)

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The crystalline title complex, $[\mathrm{Mn(C_5H_7O_2)_2(C_4H_{11}N)}]$, was prepared as a possible precursor for chemical vapour deposition of manganese(II) oxide, MnO. Two 2,4-pentanedionate and one *N*,*N*-dimethylaminoethanol ligand are coordinated to the $\mathrm{Mn^{II}}$ atom in a slightly distorted octahedral geometry. The Mn complex is isostructural with the analogous Ni, Co and Zn complexes.

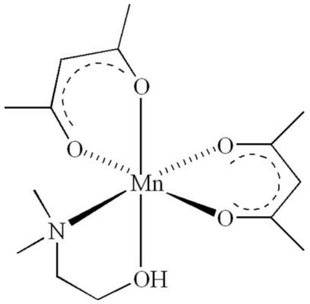
Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.096
Data-to-parameter ratio = 21.5

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

Comment

The solid-state structures of octahedral complexes of the type $[M(\mathrm{acac})_2(\mathrm{dmaeH})]$, where acac is the 2,4-pentanedionate anion and dmaeH is *N*,*N*-dimethylaminoethanol, have recently been reported for the $\mathrm{Ni^{II}}$ (Williams *et al.*, 2001; Tahir *et al.*, 2005), $\mathrm{Co^{II}}$ (Tanase *et al.*, 2004) and $\mathrm{Zn^{II}}$ derivatives (Hamid *et al.*, 2005). Via chemical vapour deposition, such complexes are potential precursors for ceramic oxides of the type *M*O (*M* = Ni, Co, Zn, *etc.*), which may be used in ceramic materials applications (Jones, 2002). The melting point of the title compound, $[\mathrm{Mn(acac)_2(dmaeH)}]$, (I) (120–125 K) is lower than that of the parent reagent $[\mathrm{Mn(acac)_2}]$ (>180 K), making (I) a promising candidate as a precursor for the deposition of thin films of MnO by aerosol-assisted chemical vapour deposition.



(I)

Compound (I) (Fig. 1) is isostructural with the analogous Co (Williams *et al.*, 2001), Ni (Tahir *et al.*, 2005; Tanase *et al.*, 2004) and Zn (Hamid *et al.*, 2005) complexes. The cell parameters are very similar: the maximum deviation for any of the angles is less than 1.4°. The cell volumes for the Mn, Ni and Zn derivatives, all measured at 100 K, are 870.3, 836.9 and 845.6 Å^3, respectively, and seem to follow the trend of the ionic radii of the cations, listed as 0.83, 0.745, 0.69 and 0.74 Å for Mn, Co, Ni and Zn, respectively (Shannon, 1976). The volume for the Co complex (Tanase *et al.*, 2004), measured at room temperature and also reported to be disordered, is 884.8 Å^3, and thus deviates from the general trend.

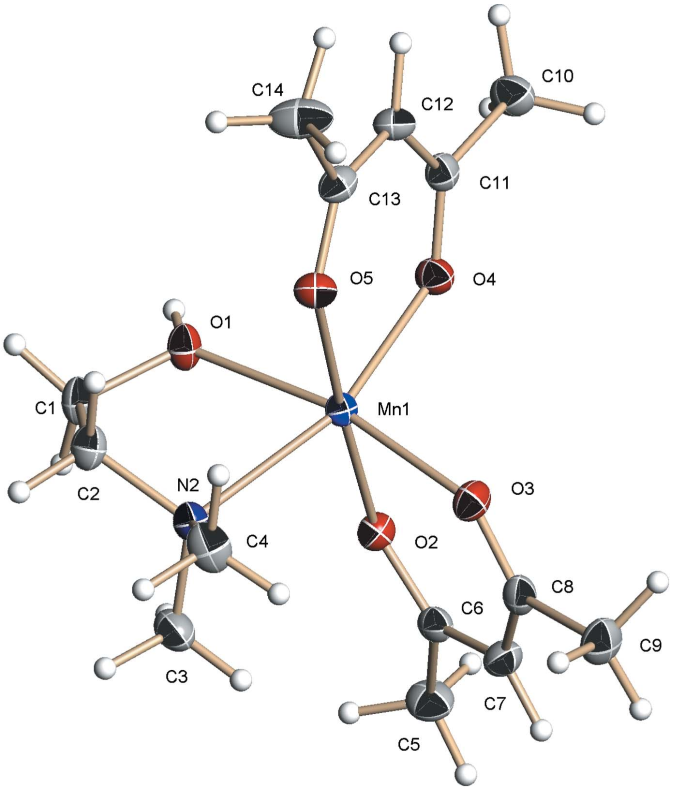


Figure 1
A view of the title complex, showing the atom-labelling scheme and with displacement ellipsoids at the 50% probability level.

The geometric environment around the central metal atom of the title complex is a distorted octahedron, with O—Mn—O 'bite' angles of 86.19 (5) and 83.37 (5)° (Table 1, Fig. 1) for the acac groups. In contrast, the dmaeH ligand, which forms a five-membered chelate ring, has a smaller O—Mn—O bite angle of only 74.67 (5)°. The Mn—O bond lengths for the acac ligands range from 2.118 (12) to 2.156 (12) Å (Table 1). These are in good agreement with the values reported for another manganese(II) bis(acetylacetonate) complex, $[Mn(acac)_2(H_2O)_2]$ (2.123 and 2.142 Å; Montgomery & Lingafelter, 1968), but slightly longer than the values reported for the manganese(III) complex $Mn(acac)_3$ (1.894–1.959 Å; Morosin *et al.*, 1964). The Mn—N bond length observed here is 2.342 (14) Å, while those for the complex $[Mn(bipy)_2(H_2O)]_2[Me_2NCH_2CO_3)][ClO_3]\cdot 2H_2O$, also octahedral, are 2.245 and 2.257 Å (Chen *et al.*, 1995).

In the crystal structure, an intermolecular O—H···O hydrogen bond (Table 2) links the molecules into centrosymmetric dimers.

Experimental

N,*N*-Dimethylaminoethanol (0.1 ml, 1.0 mmol) was added to a stirred solution of $Mn(acac)_2$ (0.254 g 1.0 mmol) in toluene (20 ml). Continuous stirring at room temperature for 4 h, followed by removal of all volatiles *in vacuo*, gave a dry powder of (I). Yellowish plate-like crystals suitable for single-crystal X-ray analysis were grown from a concentrated solution in toluene at room temperature after one week.

Crystal data

$[Mn(C_5H_7O_2)_2(C_4H_{11}N)]$
$M_r = 342.29$
Triclinic, $P\overline{1}$
$a = 7.6392\ (14)$ Å
$b = 10.5533\ (19)$ Å
$c = 11.766\ (2)$ Å
$\alpha = 105.235\ (3)^\circ$
$\beta = 103.187\ (3)^\circ$
$\gamma = 98.154\ (3)^\circ$
$V = 870.3\ (3)$ Å^3

$Z = 2$
$D_x = 1.306$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5563 reflections
$\theta = 2.3$–30.5°
$\mu = 0.78$ mm^{-1}
$T = 110\ (2)$ K
Plate, yellowish
0.37 × 0.27 × 0.02 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
$T_{min} = 0.772$, $T_{max} = 0.99$
8862 measured reflections

4296 independent reflections
3711 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 28.3^\circ$
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.096$
$S = 1.05$
4296 reflections
200 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.2295P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.63$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mn1—O3	2.1183 (12)	Mn1—O2	2.1565 (11)
Mn1—O4	2.1226 (12)	Mn1—O1	2.2354 (12)
Mn1—O5	2.1262 (12)	Mn1—N2	2.3427 (14)
O3—Mn1—O4	102.65 (5)	O5—Mn1—O1	91.31 (5)
O3—Mn1—O5	94.01 (5)	O2—Mn1—O1	91.47 (5)
O4—Mn1—O5	86.19 (5)	O3—Mn1—N2	94.92 (5)
O3—Mn1—O2	83.37 (5)	O4—Mn1—N2	162.14 (5)
O4—Mn1—O2	93.26 (5)	O5—Mn1—N2	89.40 (5)
O5—Mn1—O2	177.15 (4)	O2—Mn1—N2	91.96 (5)
O3—Mn1—O1	168.27 (5)	O1—Mn1—N2	74.67 (5)
O4—Mn1—O1	88.12 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.847 (16)	1.865 (18)	2.6777 (17)	160 (3)

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

The hydroxyl H atom was located in a difference Fourier map and was refined isotropically, with the O—H bond distance restrained to 0.90 (2) Å. All other H atoms were placed in calculated positions, with C—H = 0.95 and 0.98 Å, and were refined with $U_{iso}(H) = 1.5$ (methyl) or 1.2 (methylene) times $U_{eq}(C)$. Methyl H atoms were allowed to rotate to best fit the experimental electron density. The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small (Herbstein, 2000). The highest

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residual peak is located 0.81 Å from atom Mn1, and the deepest hole is found 1.22 Å from atom H4*C*, close to the NMe_2 group.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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