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Sodium tris(acetylacetonato-κ^2O,O')manganese(II)

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Key indicators

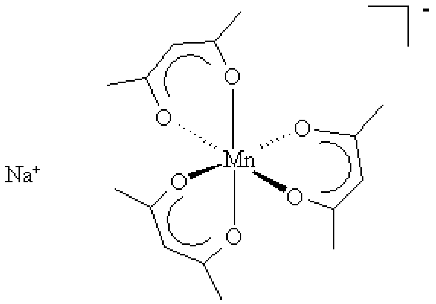
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.113
Data-to-parameter ratio = 16.9

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

In the title compound, $Na[Mn(C_5H_7O_2)_3]$, the manganese(II) ions are each coordinated by six O atoms from three different acetylacetonate ligands in a trigonal prismatic geometry. The octahedrally coordinated Na cations are sandwiched between two adjacent $[Mn(acac)_3]^-$ units (acac is acetylacetonate). The three crystallographically distinct Mn cations are each located on a crystallographic threefold rotation axis. Two Na cations are also located on special positions with site symmetry 3 and two further Na cations occupy special positions with site symmetry $\overline{3}$.

Comment

Metal–acetylacetonate complexes have been of interest as precursors in the preparations of thin films, nanoparticles and nanowires in recent years (Skordas *et al.*, 2003; Sun *et al.*, 2004; Chang & Wu, 2004). Whereas a great deal of work has been reported on the synthesis and characterization of various chromium(III), iron(III), cobalt(III) and manganese(III) complexes of the acetylacetonate ligand, there are only a few complexes reported with manganese(II) (Shibata *et al.*, 1984, 1985). Unlike the crystal structure of the title complex, the manganese(II)–acetylacetonate complexes found in the literature exist as trimers, $[Mn(acac)_2]_3$. As part of a general method for the preparation of stable manganese(II) complexes, we prepared $Na[Mn(acac)_3]$, (I), and its crystal structure is presented here (Table 1). Compound (I) was prepared at room temperature by the reaction of $Mn(acac)_3$ with $NaBH_4$ in methanol. The purpose of the $NaBH_4$ was twofold, as the reducing agent for manganese(III) and as the Na ion source for the final product.



(I)

The manganese(II) ions are each coordinated by six O atoms from three different acetylacetonate ligands in a trigonal prismatic geometry (Fig. 1). Every acetylacetonate O atom is also coordinated to an Na cation, resulting in distorted octahedral NaO_6 groupings sandwiched between two adjacent

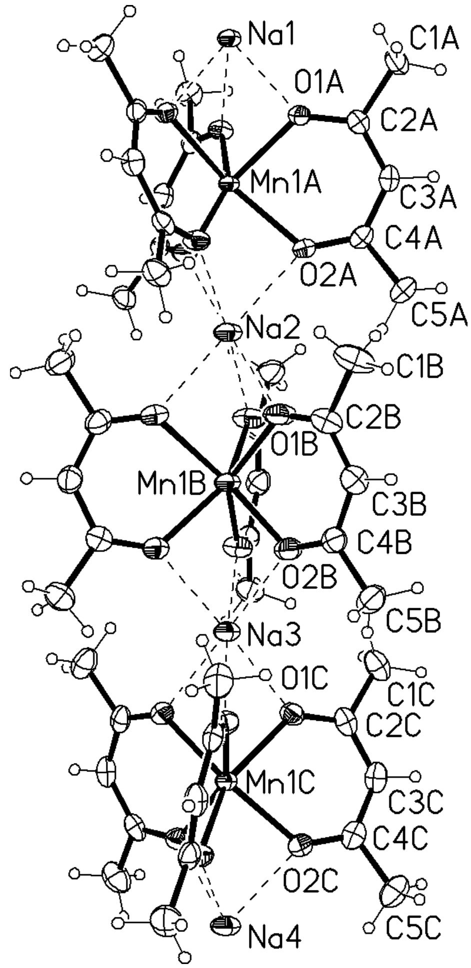


Figure 1
View of a fragment of (I), showing 50% displacement ellipsoids. H atoms are represented by spheres of arbitrary size.

$[Mn(acac)_3]^-$ units (Fig. 2). This results in infinite columns of alternating manganese- and sodium-centred polyhedra, propagating along [111] and equivalent directions. The order of cations within a column is Na1—Mn1*A*—Na2—Mn1*B*—Na3—Mn1*C*—Na4—Mn1*C*—Na3—Mn1*B*—Na2—Mn1*A*; the sequence then repeats.

Experimental

A solution of $Mn(acac)_3$ (20 mg, 0.057 mmol) dissolved in methanol (15 ml) was added to a methanol solution (5 ml) of $NaBH_4$ (3.3 mg, 0.087 mmol). After stirring the reaction mixture for an hour, the solution was concentrated by removing the solvent *in vacuo*. Slow diethyl ether diffusion into the above solution resulted in colorless prismatic crystals of $Na[Mn(acac)_3]$.

Crystal data

$Na[Mn(C_5H_7O_2)_3]$
$M_r = 375.25$
Cubic, $Pa\overline{3}$
$a = 22.075$ (3) Å
$V = 10757$ (3) Å^3
$Z = 24$
$D_x = 1.390$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 9160 reflections
$\theta = 2.3$–$26.0°$
$\mu = 0.79$ mm^{-1}
$T = 100$ (2) K
Prism, colorless
0.36 × 0.34 × 0.24 mm

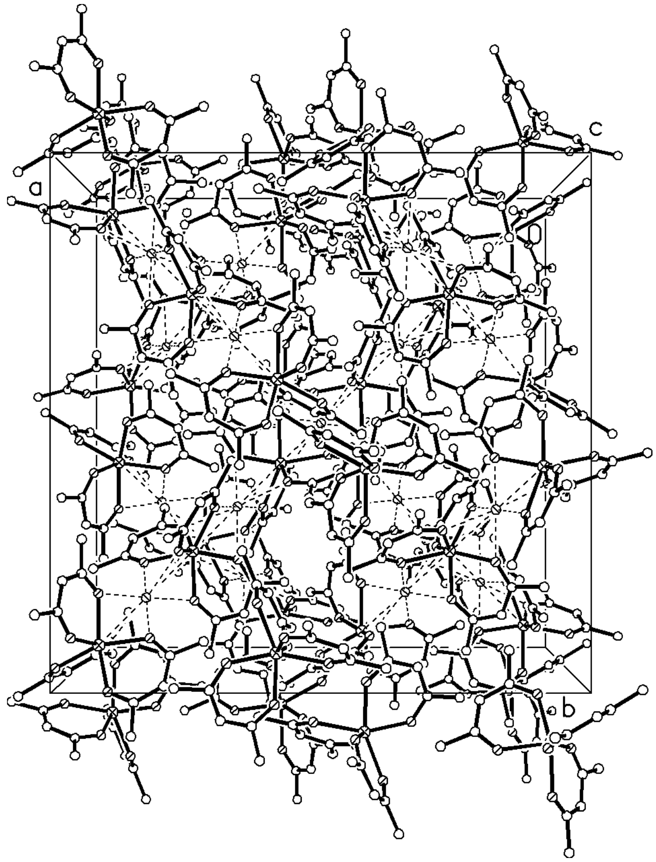


Figure 2
The packing in (I), viewed along the *c* axis. H atoms bonded to C atoms have been omitted for clarity. Na—O bonds are indicated by dashed lines.

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{min} = 0.760$, $T_{max} = 0.834$
80 828 measured reflections
3539 independent reflections
3129 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 26.0°$
$h = -27 \rightarrow 27$
$k = -27 \rightarrow 26$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.113$
$S = 1.01$
3539 reflections
209 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 6.8P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Mn1*A*—O2*A*	2.1481 (13)	Na1—O1*A*	2.4026 (13)
Mn1*A*—O1*A*	2.1493 (13)	Na2—O1*B*	2.4231 (17)
Mn1*B*—O1*B*	2.1542 (15)	Na2—O2*A*	2.4651 (16)
Mn1*B*—O2*B*	2.1690 (14)	Na3—O2*B*	2.4351 (16)
Mn1*C*—O1*C*	2.1486 (14)	Na3—O1*C*	2.4579 (16)
Mn1*C*—O2*C*	2.1562 (14)	Na4—O2*C*	2.3605 (14)

All H atoms were initially positioned geometrically. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.98 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C), but each group was allowed to rotate freely about its C—C bond. The positions of the H atoms on the central C atoms were refined as riding, with C—H = 0.95 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The torsion angle variables for the methyl groups were omitted from the last cycle of refinement after all structural parameters had converged.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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