

O(7)	1.1706 (3)	0.4473 (3)	-0.0507 (2)	4.3 (1)
O(8)	0.9112 (4)	0.5491 (3)	-0.2403 (3)	6.7 (2)
Na	0.9929 (2)	0.3781 (2)	-0.0998 (1)	4.60 (9)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Ge—O(1)	1.870 (3)	Na···O(7 ⁱ)	2.478 (3)
Ge—O(2)	1.806 (2)	O(1)—C(1)	1.428 (4)
Ge—O(3)	1.796 (2)	O(2)—C(2)	1.427 (4)
Ge—O(4)	1.866 (3)	O(3)—C(7)	1.435 (4)
Ge—O(5)	1.769 (2)	O(4)—C(8)	1.430 (4)
O(5)···Na	2.313 (3)	C(13)—O(6)	1.324 (8)
O(6)···Na	2.332 (4)	C(14)—O(7)	1.408 (5)
O(7)···Na	2.359 (3)	C(15)—O(8)	1.359 (6)
O(8)···Na	2.347 (4)		
O(1)—Ge—O(2)	87.6 (1)	C(13)—O(6)···Na	128.9 (3)
O(1)—Ge—O(3)	88.9 (1)	C(14)—O(7)···Na	130.2 (3)
O(1)—Ge—O(4)	173.5 (1)	C(14)—O(7)···Na ⁱ	112.5 (3)
O(1)—Ge—O(5)	95.9 (1)	Na···O(7)···Na ⁱ	95.7 (1)
O(2)—Ge—O(3)	123.8 (1)	C(15)—O(8)···Na	129.8 (3)
O(2)—Ge—O(4)	90.0 (1)	O(5)···Na···O(6)	87.9 (1)
O(2)—Ge—O(5)	115.6 (1)	O(5)···Na···O(7)	116.4 (1)
O(3)—Ge—O(4)	87.4 (1)	O(5)···Na···O(7 ⁱ)	82.6 (1)
O(3)—Ge—O(5)	120.6 (1)	O(5)···Na···O(8)	143.3 (1)
O(4)—Ge—O(5)	90.6 (1)	O(6)···Na···O(7)	105.8 (1)
Ge—O(1)—C(1)	110.6 (2)	O(6)···Na···O(7 ⁱ)	168.5 (1)
Ge—O(2)—C(2)	112.8 (2)	O(6)···Na···O(8)	94.2 (1)
Ge—O(3)—C(7)	113.8 (2)	O(7)···Na···O(7 ⁱ)	84.3 (1)
Ge—O(4)—C(8)	111.3 (2)	O(7)···Na···O(8)	98.3 (1)
Ge—O(5)···Na	136.3 (1)	O(7 ⁱ)···Na···O(8)	89.5 (1)

Symmetry code: (i) $2 - x, 1 - y, -z$.

The computer programs used were those of the NRCVAX package (Larson *et al.*, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1145). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Transition-Metal Antimony Ethoxides: $M_2\text{Sb}_4(\text{OEt})_{16}$; $M = \text{Ni or Mn}$

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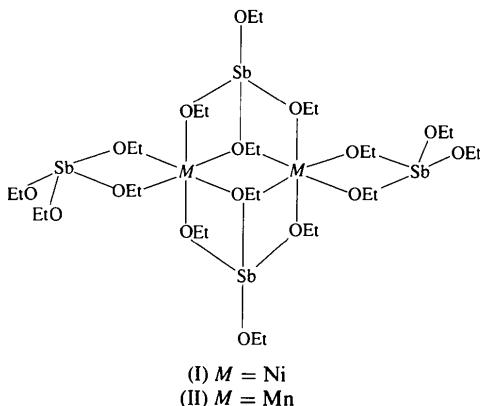
Abstract

Two new metal alkoxides, nickel antimony ethoxide, $\text{Ni}_2\text{Sb}_4(\text{OEt})_{16}$, and manganese antimony ethoxide, $\text{Mn}_2\text{Sb}_4(\text{OEt})_{16}$, have been investigated by means of single-crystal X-ray diffraction. These two transition-metal ethoxides are isostructural with $\text{Mg}_2\text{Sb}_4(\text{OEt})_{16}$. The major structural differences result mainly from the different sizes of the nickel, manganese and magnesium ions. The fourfold coordination around the Sb atoms and the sixfold coordination around the other metal ions, as well as the conformations of the ethoxide groups, are similar in all three compounds.

Comment

Metal alkoxides have become important precursors in the sol-gel route for preparation of, for example, thin oxide layers and oxide materials with special physical properties (Bradley, Mehrotra & Gaur, 1978). Extended knowledge of the molecular structures of metal alkoxides is of importance to provide the basis for understanding various physical and chemical properties, including the gelling process. The present study describes the synthesis and structural characterization of two new transition-metal antimony ethoxides with formulae $\text{Ni}_2\text{Sb}_4(\text{OEt})_{16}$, (I), and $\text{Mn}_2\text{Sb}_4(\text{OEt})_{16}$, (II). The

two structures are isostructural with the previously published alkaline-earth-metal antimony ethoxide of composition $Mg_2Sb_4(OEt)_{16}$ (Bemm, Lashgari, Norrestam, Nygren & Westin, 1993). This investigation is a part of research into heterometallic alkoxide complexes and their applications for the syntheses of new oxide materials, and is particularly focused towards complexes containing antimony.



The molecular structures include a tetrameric fragment with an $Sb_2M_2O_6$ skeleton formed by the atoms Sb(1), M , O(1), O(2), O(3) and their symmetry equivalents. Such fragments are frequently found in alkoxides and related compounds. Two ethoxy coordinated Sb atoms are attached to the fragment via two μ -O atom bridges to form the entire molecule (Fig. 1 and 2). The Sb atoms are coordinated by four O atoms. The coordination polyhedron can be considered to be a distorted trigonal bipyramidal where the lone pair of Sb occupies one of the axial positions. The other metal ions (Ni^{2+} or Mn^{2+}) have approximately octahedral environments and are coordinated by O atoms.

The differences in bond lengths and bond angles, including those of isostructural $Mg_2Sb_4(OEt)_{16}$ (Bemm, Lashgari, Norrestam, Nygren & Westin, 1993), are mainly the result of the differences in the sizes of the three metal ions Ni^{2+} , Mn^{2+} and Mg^{2+} . The M —O bond length averages, 2.08(4), 2.19(6) and 2.10(5) Å, respectively, are linearly correlated to the ionic radii (Shannon, 1976). The differences in Sb—O bond lengths in $Ni_2Sb_4(OEt)_{16}$, $Mn_2Sb_4(OEt)_{16}$ and $Mg_2Sb_4(OEt)_{16}$ are negligible: the Sb(1)—O bond averages are 2.08(14), 2.08(14) and 2.08(15) Å, respectively, and the Sb(2)—O distances are 2.05(8), 2.06(10) and 2.06(10) Å, respectively. The average bond lengths O— C_α , 1.44(2), 1.43(3) and 1.43(3) Å, and C_α — C_β , 1.50(4), 1.49(4) and 1.48(3) Å, in the ethoxide groups differ only within the e.s.d.'s, and can thus be considered as equal. A more detailed discussion on common structural features of crystals of these compounds is given in the paper on $Mg_2Sb_4(OEt)_{16}$ (Bemm, Lashgari, Norrestam, Nygren & Westin, 1993).

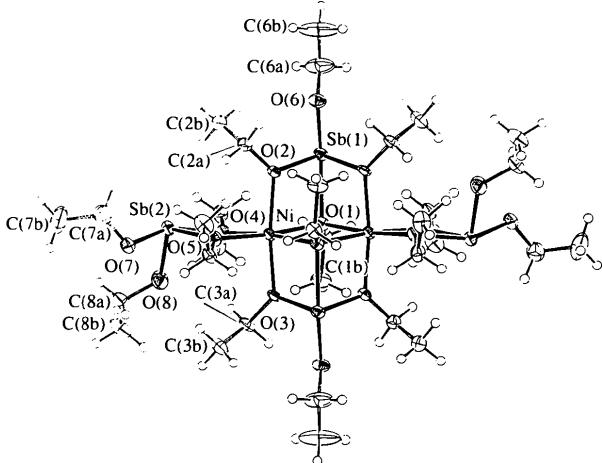


Fig. 1. ORTEPII (Johnson, 1976) drawing (displacement ellipsoids are drawn at 75% probability levels) of the molecular structure of $Ni_2Sb_4(OEt)_{16}$, almost perpendicular to the plane of the metal atoms. Unlabelled atoms indicate equivalent atoms related by inversion symmetry to the labelled ones.

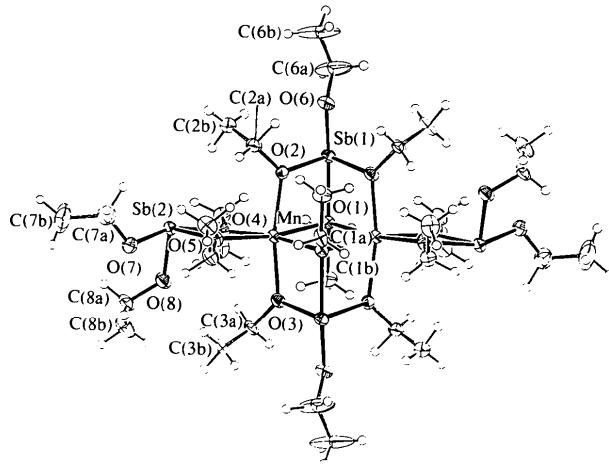


Fig. 2. ORTEPII drawing (displacement ellipsoids are drawn at the 75% probability level) of the molecular structure of $Mn_2Sb_4(OEt)_{16}$, almost perpendicular to the plane of the metal atoms. Unlabelled atoms indicate equivalent atoms related by inversion symmetry to the labelled ones.

Experimental

The syntheses of the two transition metal alkoxides, $Ni_2Sb_4(OEt)_{16}$ and $Mn_2Sb_4(OEt)_{16}$, were performed in a similar way to that of $Ni_5Sb_3O_2(OEt)_{15}(HOEt)_4$ (Bemm, Norrestam, Nygren & Westin, 1992) from $NiCl_2$ and $MnCl_2$, but with pure ethanol as solute and in higher concentrations. The composition ratios $NaSb(OEt)_4:NiCl_2$ and $NaSb(OEt)_4:MnCl_2$ were 2:1 and the solutions produced were green and light pink, respectively. From the evaporated solution, transpar-

ent needle-shaped Mn₂Sb₄(OEt)₁₆ crystals were obtained. The green needle-shaped Ni₂Sb₄(OEt)₁₆ crystals were grown from an ethanol-toluene (1:2) solution.

Compound (I)

Crystal data



$M_r = 1325.34$

Triclinic

$P\bar{1}$

$a = 9.093 (7) \text{ \AA}$

$b = 12.108 (9) \text{ \AA}$

$c = 12.599 (8) \text{ \AA}$

$\alpha = 64.10 (4)^\circ$

$\beta = 80.35 (5)^\circ$

$\gamma = 87.61 (6)^\circ$

$V = 1229.4 (15) \text{ \AA}^3$

$Z = 1$

$D_x = 1.790 (3) \text{ Mg m}^{-3}$

Data collection

Stoe four-circle diffractometer

ω -2 θ scans

Absorption correction:
by integration from crystal
shape

$T_{\min} = 0.454, T_{\max} = 0.533$

$4656 \text{ measured reflections}$

$4355 \text{ independent reflections}$

Refinement

Refinement on F

$R = 0.033$

$wR = 0.038$

3241 reflections

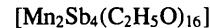
368 parameters

Only coordinates of H atoms
refined

$w = 1/[\sigma^2(F_o) + (0.0002|F^2|)]$

Compound (II)

Crystal data



$M_r = 1317.84$

Triclinic

$P\bar{1}$

$a = 9.143 (4) \text{ \AA}$

$b = 12.318 (4) \text{ \AA}$

$c = 12.798 (4) \text{ \AA}$

$\alpha = 63.65 (2)^\circ$

$\beta = 79.90 (2)^\circ$

$\gamma = 86.86 (2)^\circ$

$V = 1271.1 (8) \text{ \AA}^3$

$Z = 1$

$D_x = 1.722 (2) \text{ Mg m}^{-3}$

Data collection

Stoe four-circle diffractometer

ω -2 θ scans

Absorption correction:
by integration from crystal
shape

$T_{\min} = 0.353, T_{\max} = 0.557$

$4770 \text{ measured reflections}$

$4463 \text{ independent reflections}$

3935 observed reflections

$[|F_o| > 3\sigma(|F_o|)]$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 25.0^\circ$

$h = 0 \rightarrow 11$

$k = -15 \rightarrow 15$

$l = -15 \rightarrow 15$

3 standard reflections

frequency: 240 min
intensity decay: 4.1%

Refinement

Refinement on F

$R = 0.039$

$wR = 0.054$

3935 reflections

368 parameters

Only coordinates of H atoms
refined

$w = 1/[\sigma^2(F_o) + (0.0002|F^2|)]$

$(\Delta/\sigma)_{\max} = 0.25$

$\Delta\rho_{\max} = 1.8 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -1.7 \text{ e \AA}^{-3}$

Extinction correction: none

Atomic scattering factors
from *International Tables*
for X-ray Crystallography
(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	x	y	z	U_{eq}
[Ni ₂ Sb ₄ (C ₂ H ₅ O) ₁₆]				
Sb(1)	0.16692 (5)	0.20025 (4)	-0.20959 (4)	0.0233 (2)
Sb(2)	-0.05941 (5)	0.28055 (4)	0.19646 (4)	0.0284 (2)
Ni(1)	-0.02947 (10)	0.08912 (8)	0.06777 (7)	0.0213 (3)
O(1)	-0.0477 (5)	0.0859 (4)	-0.0945 (4)	0.023 (2)
O(2)	0.1642 (5)	0.1839 (4)	-0.0445 (4)	0.026 (3)
O(3)	-0.2108 (5)	-0.0262 (4)	0.1725 (4)	0.024 (2)
O(4)	-0.1236 (5)	0.2471 (4)	0.0535 (4)	0.026 (2)
O(5)	0.0059 (5)	0.1097 (4)	0.2202 (4)	0.026 (2)
O(6)	0.3855 (5)	0.2393 (4)	-0.2415 (4)	0.031 (2)
O(7)	-0.0386 (6)	0.2363 (5)	0.3684 (4)	0.037 (2)
O(8)	-0.2744 (6)	0.2553 (5)	0.2507 (5)	0.052 (3)
C(1a)	-0.1718 (8)	0.1399 (7)	-0.1560 (6)	0.030 (3)
C(1b)	-0.3207 (9)	0.0847 (7)	-0.0866 (7)	0.036 (3)
C(2a)	0.2747 (8)	0.2434 (7)	-0.0144 (7)	0.035 (3)
C(2b)	0.2661 (10)	0.3812 (8)	-0.0732 (8)	0.050 (4)
C(3a)	-0.3515 (8)	0.0105 (7)	0.2234 (6)	0.030 (3)
C(3b)	-0.3658 (10)	-0.0368 (8)	0.3587 (7)	0.046 (4)
C(4a)	-0.1476 (9)	0.3573 (7)	-0.0465 (7)	0.033 (3)
C(4b)	-0.3116 (10)	0.3768 (8)	-0.0524 (8)	0.048 (4)
C(5a)	0.0321 (8)	0.0153 (7)	0.3330 (6)	0.028 (3)
C(5b)	0.1924 (10)	0.0139 (8)	0.3487 (7)	0.043 (4)
C(6a)	0.4426 (12)	0.3406 (10)	-0.3529 (8)	0.064 (5)
C(6b)	0.5993 (13)	0.3602 (12)	-0.3718 (11)	0.082 (6)
C(7a)	0.0785 (10)	0.2958 (9)	0.3858 (8)	0.050 (4)
C(7b)	0.0192 (15)	0.3855 (11)	0.4346 (11)	0.096 (6)
C(8a)	-0.3516 (9)	0.2821 (8)	0.3446 (7)	0.041 (4)
C(8b)	-0.5085 (10)	0.3092 (9)	0.3235 (8)	0.048 (4)
[Mn ₂ Sb ₄ (C ₂ H ₅ O) ₁₆]				
Sb(1)	0.17895 (5)	0.20246 (4)	-0.21597 (4)	0.0239 (2)
Sb(2)	-0.06175 (5)	0.28507 (4)	0.19934 (4)	0.0288 (2)
Mn(1)	-0.03136 (10)	0.09262 (8)	0.06834 (8)	0.0224 (4)
O(1)	-0.0392 (5)	0.0959 (4)	-0.1063 (4)	0.023 (2)
O(2)	0.1765 (5)	0.1857 (4)	-0.0533 (4)	0.030 (2)
O(3)	-0.2191 (5)	-0.0288 (4)	0.1775 (4)	0.029 (2)
O(4)	-0.1195 (5)	0.2569 (4)	0.0527 (4)	0.029 (2)
O(5)	0.0061 (5)	0.1174 (4)	0.2225 (4)	0.028 (2)
O(6)	0.3977 (5)	0.2393 (4)	-0.2472 (4)	0.031 (2)
O(7)	-0.0433 (6)	0.2410 (4)	0.3701 (4)	0.042 (2)
O(8)	-0.2739 (5)	0.2542 (5)	0.2533 (5)	0.045 (2)
C(1a)	-0.1631 (7)	0.1523 (6)	-0.1648 (6)	0.031 (3)

C(1b)	-0.3078 (8)	0.0914 (7)	-0.0976 (7)	0.038 (3)
C(2a)	0.2843 (8)	0.2441 (7)	-0.0247 (6)	0.036 (3)
C(2b)	0.2698 (10)	0.3808 (8)	-0.0809 (8)	0.052 (4)
C(3a)	-0.3585 (8)	0.0082 (7)	0.2256 (6)	0.033 (3)
C(3b)	-0.3737 (9)	-0.0341 (8)	0.3594 (7)	0.046 (4)
C(4a)	-0.1535 (8)	0.3664 (6)	-0.0428 (6)	0.033 (3)
C(4b)	-0.3175 (10)	0.3758 (8)	-0.0447 (8)	0.053 (4)
C(5a)	0.0298 (8)	0.0229 (7)	0.3358 (6)	0.034 (3)
C(5b)	0.1901 (9)	0.0172 (8)	0.3497 (7)	0.046 (4)
C(6a)	0.4600 (15)	0.3257 (14)	-0.3575 (9)	0.100 (7)
C(6b)	0.5930 (15)	0.3845 (14)	-0.3691 (12)	0.095 (7)
C(7a)	0.0741 (10)	0.2969 (8)	0.3864 (8)	0.058 (4)
C(7b)	0.0191 (15)	0.3870 (12)	0.4341 (12)	0.115 (7)
C(8a)	-0.3580 (8)	0.2778 (7)	0.3456 (7)	0.041 (3)
C(8b)	-0.5115 (9)	0.3076 (9)	0.3212 (8)	0.057 (4)

Table 2. Selected geometric parameters (\AA , °)

[Ni ₂ Sb ₄ (C ₂ H ₅ O) ₁₆]	[Mn ₂ Sb ₄ (C ₂ H ₅ O) ₁₆]
Sb(1)—O(1)	2.313 (4)
Sb(1)—O(2)	1.996 (4)
Sb(1)—O(3')	1.987 (4)
Sb(1)—O(6)	1.995 (5)
Sb(2)—O(4)	2.182 (5)
Sb(2)—O(5)	2.034 (4)
Sb(2)—O(7)	2.033 (5)
Sb(2)—O(8)	1.950 (5)
Ni(1)—O(1)	2.097 (4)
Ni(1)—O(2)	2.082 (5)
Ni(1)—O(3)	2.066 (4)
Ni(1)—O(4)	2.010 (4)
Ni(1)—O(5)	2.117 (4)
O(1)—C(1a)	1.446 (8)
O(2)—C(2a)	1.449 (8)
O(3)—C(3a)	1.474 (8)
O(4)—C(4a)	1.420 (8)
O(5)—C(5a)	1.434 (8)
O(6)—C(6a)	1.431 (10)
O(7)—C(7a)	1.407 (10)
O(8)—C(8a)	1.427 (9)
O(1)—Sb(1)—O(2)	73.9 (2)
O(1)—Sb(1)—O(3')	73.7 (2)
O(1)—Sb(1)—O(6)	153.1 (2)
O(2)—Sb(1)—O(3')	96.3 (2)
O(2)—Sb(1)—O(6)	87.7 (2)
O(3')—Sb(1)—O(6)	89.4 (2)
O(4)—Sb(2)—O(5)	72.2 (2)
O(4)—Sb(2)—O(7)	154.9 (2)
O(4)—Sb(2)—O(8)	80.3 (2)
O(5)—Sb(2)—O(7)	89.5 (2)
O(5)—Sb(2)—O(8)	102.4 (2)
O(7)—Sb(2)—O(8)	87.4 (2)
O(1)—Ni—O(1')	77.9 (2)
O(1)—Ni—O(2)	77.0 (2)
O(1')—Ni—O(2)	95.0 (2)
O(1')—Ni—O(3)	98.0 (2)
O(1')—Ni—O(4)	76.9 (2)
O(1')—Ni—O(4)	103.5 (2)
O(1')—Ni—O(5)	173.9 (2)
O(1')—Ni—O(5)	173.5 (2)
O(1')—Ni—O(5)	105.2 (2)
O(2)—Ni—O(3)	171.3 (2)
O(2)—Ni—O(4)	91.1 (2)
O(2)—Ni—O(5)	97.0 (2)
O(3)—Ni—O(4)	97.0 (2)
O(3)—Ni—O(5)	88.3 (2)
O(4)—Ni—O(5)	74.0 (2)
O(1)—Sb(1)—O(2)	74.0 (2)
O(1)—Sb(1)—O(3')	154.8 (2)
O(1)—Sb(1)—O(6)	96.2 (2)
O(2)—Sb(1)—O(3')	87.2 (2)
O(3')—Sb(1)—O(6)	90.4 (2)
O(4)—Sb(2)—O(5)	72.8 (2)
O(4)—Sb(2)—O(7)	156.6 (2)
O(4)—Sb(2)—O(8)	80.7 (2)
O(5)—Sb(2)—O(7)	89.9 (2)
O(5)—Sb(2)—O(8)	100.5 (2)
O(7)—Sb(2)—O(8)	87.3 (2)
O(1)—Mn—O(1')	80.4 (2)
O(1)—Mn—O(2)	72.4 (2)
O(1')—Mn—O(2)	96.4 (2)
O(1')—Mn—O(3)	100.6 (2)
O(1')—Mn—O(3)	72.5 (2)
O(1)—Mn—O(4)	103.9 (2)
O(1')—Mn—O(4)	172.2 (2)
O(1)—Mn—O(5)	169.5 (2)
O(1')—Mn—O(5)	105.0 (2)
O(2)—Mn—O(3)	168.0 (2)
O(2)—Mn—O(4)	91.1 (2)
O(2)—Mn—O(5)	97.7 (2)
O(3)—Mn—O(4)	100.2 (2)
O(3)—Mn—O(5)	89.7 (2)
O(4)—Mn—O(5)	71.9 (2)

The structure solution and the structural refinements were performed using SHELXS86 (Sheldrick, 1985) and SHELX76 (Sheldrick, 1976). Non-H atoms were refined with anisotropic displacement parameters, H atoms were refined isotropically with bond constraints of 1.00 \AA between the C and H atoms. Geometric calculations of bond distances and bond angles were performed with PLATON (Spek, 1990). Molecular graphics were produced using ORTEPII (Johnson, 1976).

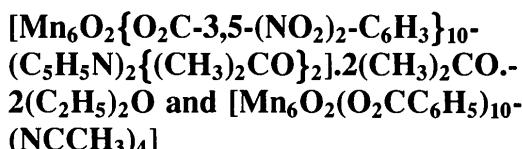
Our study of metal alkoxides is financially supported by the Swedish National Science Research Council.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the IUCr (Reference: AB1151). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Abstract

The structures of di(acetone)tetrakis(μ^3 -3,5-dinitrobenzoato- $\kappa^2 O:\kappa O'$)hexakis(μ -3,5-dinitrobenzoato- $\kappa O:\kappa O'$)-di- μ^4 -oxo-di(pyridine)tetramanganese(II)dimanganese(III)-acetone-diethyl ether (1/2/2), (1), and tetrakis(acetonitrile)tetrakis(μ^3 -benzoato- $\kappa^2 O:\kappa O'$)hexakis(μ -benzoato- $\kappa O:\kappa O'$)-di- μ^4 -oxo-tetramanganese(II)dimanganese(III), (2), are reported. Both compounds contain six octahedrally coordinated Mn centres, arranged as two Mn^{II}Mn^{III}(μ^4 -O) tetrahedra sharing the Mn^{III}–Mn^{III} edge.