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$[(Mg_{0.5},Mn_{0.5})_4\{\mu_3,\eta^2\text{-}OCH_2\overline{CH(CH_2)_3O}\}_4(EtOH)_4Cl_4].0.5EtOH$
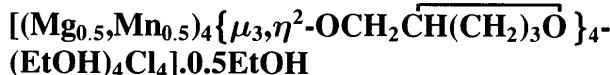
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Abstract

The reaction of $MnCl_2$ with $[Mg\{OCH_2\overline{CH(CH_2)_3O}\}_2]$ in EtOH results in a mixed Mg^{II}/Mn^{II} tetranuclear complex, tetrakis(μ_3-tetrahydrofuran-2-ylmethanolato-$O,O':O':O'$)tetrakis{chloro(ethoxo)[manganese(II),-magnesium(II)]} hemiethanol solvate, $[(Mg_{0.5},Mn_{0.5})_4Cl_4(C_2H_6O)_4(C_5H_9O_2)_4].0.5C_2H_6O$, in which all metal sites have distorted octahedral coordination spheres and metal ions together with alkoxo O atoms form a cubane-like framework.

Comment

This work is a part of our systematic study on complexes with O-atom donor functions in bidentate alkoxo ligands such as 2-tetrahydrofurfuryl alcohol

(thffo). The structures and properties of magnesium(II) (Sobota, Utko, Janas & Szafert, 1996) and vanadium(III)–magnesium(II) (Janas, Sobota, Kasprzak & Głowiak, 1997) compounds have been reported previously. In this paper, we describe the structure of a magnesium(II)/manganese(II)–alkoxide complex, (I).

$M = Mg,Mn$

(I)
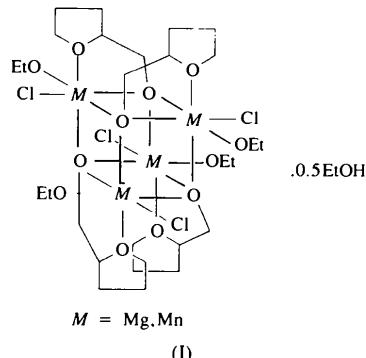
The crystal structure of (I) comprises discrete complex molecules and an ethanol molecule of crystallization. The complex consists of four crystallographically independent distorted octahedral metal sites with shared common edges. Magnesium(II) and manganese(II) ions are distributed in equal proportions among all the sites. The coordination sphere of each metal ion is formed by Cl^- anions, ethanol molecules and a chelating tetrahydrofuran-2-ylmethanolato ligand. On the other hand, the metal ions and alkoxo O atoms form a distorted cubane-like framework (Fig. 1 and Table 2) due to steric requirements resulting from the formation of five-membered chelate rings. The M—μ-O bonds (where $M = Mn^{II}/Mg^{II}$) of 2.106 (2)–2.191 (2) Å are comparable with M—μ_3-O distances found in magnesium(II) compounds: 1.934 (3)–2.226 (3) Å in $[Mg_4\{\mu_3,\eta^2\text{-}OCH_2CH(CH_2)_3O\}_2\{\mu,\eta^2\text{-}OCH_2CH(CH_2)_3O\}_4Cl_2]$ (Sobota, Utko, Janas & Szafert, 1996) and 2.060 (1)–2.083 (2) Å in $[Mg_4(\mu_3\text{-}EtOH)_4(C_5H_5)_4]$ (Lehmkuhl, Mehler, Benn, Rufinski & Kruger, 1986). The changes of M—μ-O bond lengths are reflected in the intramolecular $M\cdots M$ distances. Two cubane-face diagonal vectors $M(1)\cdots M(2)$ and $M(3)\cdots M(4)$ are longer by ca 0.145 (3) Å than the other four (Table 2). The M—O(ether) bond lengths in the title compound change from 2.145 (2) to 2.193 (2) Å. In the structure of the octahedral magnesium(II) $[MgCl(thf)_5]^+$ cation (Sobota, Płuzinski, Utko & Lis, 1989) and the manganese(II) $[Mn\{OCH_2CH(CH_2)_3O\}_2Cl_2]$ (Sobota, Utko & Jerzykiewicz, 1997) complex, the ether O atoms are 2.156 (4) and 2.223 (2) Å away from the metal ion, respectively. The ethanol ligands give a relatively long M—O distance of 2.231 (2) Å, probably because of the intramolecular hydrogen bonds in which they are involved. Every coordinated hydroxyl group forms a strong hydrogen bond with the chlorine anion bonded to a neighbouring metal ion (Table 3). The average M—Cl

distance of 2.383 (5) Å is similar to those found in the magnesium(II) [Mg$_4$\{μ_3,η^2-OCH$_2$CH(CH$_2$)$_3$O\}$_2$\{μ,η^2-OCH$_2$CH(CH$_2$)$_3$O\}$_4$Cl$_2$] (Sobota, Utko, Janas & Szafert, 1996) and [MgCl(thf)$_5$]$^+$ [2.384 (9) Å] (Sobota, Pluzinski & Utko, 1989)] compounds, and is shorter than those in a manganese(II) compound [Mn$_2$(C$_6$H$_{13}$N$_2$)$_2$Cl$_6$] [2.372 (4) Å] (Viossat & Rodier, 1988). The uncoordinated disordered ethanol molecule, which is involved in an intermolecular hydrogen bond (Table 3), is found to be trapped in the cavities formed by the molecules of the complex.
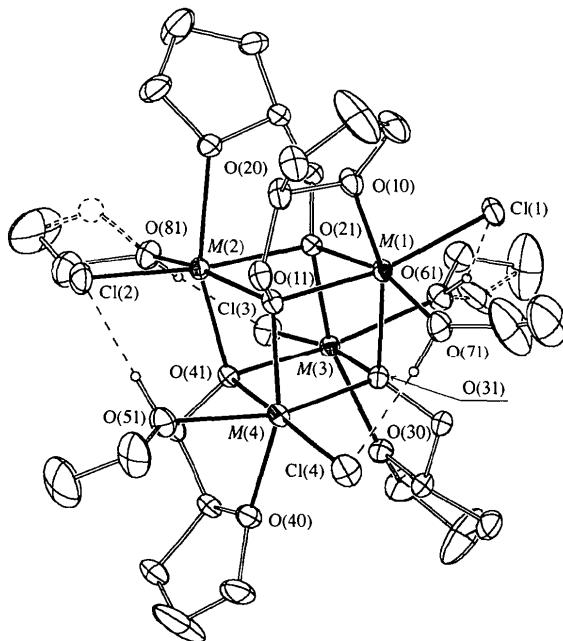

Fig. 1. The molecular structure of the title complex with the atom-numbering scheme. Displacement ellipsoids are drawn at the 25% probability level. The C-bonded H atoms have been omitted for clarity. The C and H atoms are represented by circles of arbitrary radii. Intramolecular hydrogen bonds and the disordered region are shown as dashed lines; $M(x)$ = Mn(x)/Mg(x).

Some inorganic mixed Mg/Mn site compounds have been reported (Norrestam & Bovin, 1987; Utzolino & Bluhms, 1996) and cubane-type structures are known for both ions: [Mn$_4$O$_3$Cl$_4$(O$_2$CR)$_3$(py)$_3$] (Wemple, Tsai, Folting, Henderickson & Christou, 1993), [Mn$_4$(OEt)$_4$(EtOH)$_2$(DPM)$_4$] (Taft, Caneschi, Pence, Delfs, Papaefthymiou & Lippard, 1993) and [Mg$_4$(μ_3-EtOH)$_4$(C$_5$H$_5$)] (Lehmkuhl et al., 1986) (in the case of manganese cubes, the metal atom was tri- or tetravalent).

Experimental

The air- and moisture-sensitive title compound was prepared under dried N$_2$. A mixture of 1.26 g (10 mmol) MnCl$_2$ and 2.26 g (10 mmol) [Mg(OCH$_2$CH(CH$_2$)$_3$O)$_2$] in 50 ml

EtOH was refluxed for 12 h until the solution cleared. The resultant solution was filtered and left to crystallize at room temperature. After 24 h, the colourless needle-shaped crystals were filtered off and washed with n-hexane (3 × 5 ml). Yield 2.36 g (53%). Found: Cl 15.28, Mg 5.27, Mn 12.03%; requires: Cl 15.39, Mg 5.28, Mn 12.09%. IR (Nujol, cm^{-1}): 225 (w), 255 (w), 374 (s), 404 (vs), 448 (s), 530 (s, br), 590 (s), 612 (w), 628 (w), 660 (w), 720 (s), 810 (s), 842 (w), 876 (s), 917(m), 940 (m), 960 (m), 1010 (s), 1044 (vs), 1060 (sh), 1080 (sh), 1135 (w), 1150 (w), 1270 (w), 1300 (w), 1640 (w, br), 3300 (vs, br). The crystal was sealed in a glass capillary under a nitrogen stream.

Crystal data

[(Mg$_{0.5}$,Mn$_{0.5}$)$_4$Cl$_4$(C$_2$H$_6$O)$_4$-(C$_5$H$_9$O$_2$)$_4$].0.5C$_2$H$_6$O	Mo $K\alpha$ radiation
M_r = 1823.18	λ = 0.71073 Å
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
a = 12.079 (3) Å	θ = 9–15°
b = 12.150 (3) Å	μ = 0.893 mm^{-1}
c = 17.641 (4) Å	T = 300 (1) K
α = 87.02 (3)°	Block
β = 78.00 (3)°	0.5 × 0.4 × 0.4 mm
γ = 60.87 (3)°	Colourless
V = 2208.4 (7) Å^3	
Z = 2	
D_x = 1.371 Mg m^{-3}	
D_m = 1.376 Mg m^{-3}	
D_m measured by flotation in toluene/1,2-dibromo-methane	

Data collection

Kuma KM-4 automatic diffractometer	R_{int} = 0.067
Profile data from $\omega/2\theta$ scans	θ_{max} = 26°
Absorption correction: none	h = −10 → 10
12 510 measured reflections	k = −13 → 13
6799 independent reflections	l = −20 → 20
5692 reflections with	3 standard reflections
$I > 2\sigma(I)$	every 100 reflections
	intensity decay: 5%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = −0.13
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$wR(F^2)$ = 0.120	$\Delta\rho_{min}$ = −0.46 e Å^{-3}
6797 reflections	Extinction correction: none
476 parameters	Scattering factors from
H atoms: see below	International Tables for
w = 1/[$\sigma^2(F_o^2)$ + (0.054P)^2 + 1.6258P]	Crystallography (Vol. C)
where P = (F_o^2 + 2F_c^2)/3	

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Mn(1)†	−0.05760 (5)	0.33053 (6)	0.26370 (3)	0.0448 (2)
Mn(2)†	0.17397 (6)	0.04967 (6)	0.30612 (3)	0.0456 (2)
Mn(3)†	0.18292 (6)	0.14363 (5)	0.13191 (3)	0.0436 (2)

Mn(4)†	0.22389 (6)	0.28701 (6)	0.27267 (3)	0.0479 (2)
Mg(1)†	−0.05760 (5)	0.33053 (6)	0.26370 (3)	0.0448 (2)
Mg(2)†	0.17397 (6)	0.04967 (6)	0.30612 (3)	0.0456 (2)
Mg(3)†	0.18292 (6)	0.14363 (5)	0.13191 (3)	0.0436 (2)
Mg(4)†	0.22389 (6)	0.28701 (6)	0.27267 (3)	0.0479 (2)
Cl(1)	−0.22116 (8)	0.38968 (9)	0.19068 (5)	0.0686 (2)
Cl(2)	0.27566 (10)	−0.02245 (10)	0.41446 (5)	0.0788 (3)
Cl(3)	0.30271 (9)	−0.06203 (8)	0.06795 (5)	0.0715 (3)
Cl(4)	0.15490 (11)	0.50102 (9)	0.30670 (7)	0.0852 (3)
O(10)	−0.1741 (2)	0.3323 (2)	0.37692 (12)	0.0671 (6)
O(11)	0.0782 (2)	0.2512 (2)	0.33920 (10)	0.0492 (5)
O(20)	0.0152 (2)	0.0074 (2)	0.34697 (12)	0.0623 (6)
O(21)	0.0487 (2)	0.1306 (2)	0.22427 (11)	0.0448 (4)
O(30)	0.2657 (2)	0.2357 (2)	0.05142 (11)	0.0561 (5)
O(31)	0.1041 (2)	0.3278 (2)	0.18687 (10)	0.0448 (4)
O(40)	0.4125 (2)	0.2378 (2)	0.20108 (14)	0.0605 (6)
O(41)	0.2936 (2)	0.1001 (2)	0.22306 (11)	0.0443 (4)
O(51)	0.3280 (3)	0.2022 (3)	0.36841 (15)	0.0746 (7)
O(61)	0.0246 (2)	0.2165 (2)	0.06576 (13)	0.0634 (6)
O(71)	−0.1106 (3)	0.5256 (2)	0.2974 (2)	0.0754 (7)
O(81)	0.2804 (3)	−0.1349 (2)	0.2420 (2)	0.0723 (7)
C(11)	−0.1049 (3)	0.2905 (4)	0.4402 (2)	0.0670 (10)
C(12)	−0.2027 (4)	0.3738 (5)	0.5102 (2)	0.0939 (15)
C(13)	−0.3236 (5)	0.4219 (9)	0.4904 (4)	0.196 (5)
C(14)	−0.3098 (4)	0.3969 (6)	0.4077 (3)	0.106 (2)
C(15)	0.0151 (3)	0.3010 (3)	0.4164 (2)	0.0622 (9)
C(21)	−0.0773 (3)	0.0428 (4)	0.2976 (2)	0.0619 (8)
C(22)	−0.1124 (5)	−0.0620 (5)	0.3030 (3)	0.0869 (13)
C(23)	−0.0694 (7)	−0.1292 (6)	0.3713 (3)	0.126 (2)
C(24)	0.0004 (6)	−0.0777 (6)	0.4007 (3)	0.112 (2)
C(25)	−0.0138 (3)	0.0594 (3)	0.2183 (2)	0.0541 (7)
C(31)	0.2233 (3)	0.3661 (3)	0.0714 (2)	0.0566 (8)
C(32)	0.3244 (5)	0.2075 (5)	−0.0292 (2)	0.0919 (13)
C(33)	0.3073 (10)	0.3221 (7)	−0.0620 (3)	0.200 (4)
C(34)	0.2180 (4)	0.4299 (4)	−0.0050 (2)	0.0861 (12)
C(35)	0.0964 (3)	0.4165 (3)	0.1298 (2)	0.0524 (7)
C(41)	0.4846 (3)	0.1121 (3)	0.1633 (2)	0.0561 (8)
C(42)	0.6256 (3)	0.0716 (4)	0.1619 (3)	0.0785 (11)
C(43)	0.4970 (4)	0.2884 (5)	0.2016 (4)	0.101 (2)
C(44)	0.6269 (6)	0.1839 (7)	0.1773 (8)	0.241 (6)
C(45)	0.4313 (3)	0.0329 (3)	0.2072 (2)	0.0515 (7)
C(51)	0.3611 (7)	0.2486 (6)	0.4247 (4)	0.139 (2)
C(52)	0.4721 (9)	0.1744 (9)	0.4489 (6)	0.222 (5)
C(61)‡	−0.0029 (12)	0.1517 (7)	0.0138 (6)	0.083 (3)
C(611)§	0.028 (2)	0.199 (5)	−0.014 (2)	0.117 (11)
C(62)	−0.0436 (12)	0.2057 (7)	−0.0491 (5)	0.236 (6)
C(71)	−0.2041 (8)	0.6394 (7)	0.2858 (8)	0.249 (7)
C(72)	−0.2811 (10)	0.7340 (8)	0.3135 (8)	0.242 (6)
C(81)¶	0.3922 (11)	−0.2501 (8)	0.2565 (6)	0.112 (4)
C(811)††	0.2982 (13)	−0.2545 (10)	0.2594 (6)	0.083 (4)
C(82)	0.4084 (9)	−0.3535 (7)	0.2522 (9)	0.292 (8)
O(1E)†	0.522 (3)	0.334 (3)	0.012 (2)	0.51 (2)
C(1E)†	0.537 (3)	0.445 (2)	0.0105 (15)	0.221 (9)
C(2E)†	0.425 (3)	0.569 (3)	0.0518 (18)	0.328 (18)

† Site occupancy = 0.50.   ‡ Site occupancy = 0.74 (3).   § Site occupancy = 0.26 (3).   ¶ Site occupancy = 0.58 (2).   †† Site occupancy = 0.42 (2).

Table 2. Selected bond distances (Å)

M(1)—Cl(1)†	2.379 (2)	M(3)—O(31)	2.150 (2)
M(2)—Cl(2)	2.384 (2)	M(4)—O(31)	2.183 (2)
M(3)—Cl(3)	2.389 (2)	M(2)—O(41)	2.121 (2)
M(4)—Cl(4)	2.379 (2)	M(3)—O(41)	2.184 (2)
M(1)—O(10)	2.185 (2)	M(4)—O(41)	2.161 (2)
M(2)—O(20)	2.193 (2)	M(1)—O(71)	2.212 (3)
M(3)—O(30)	2.145 (2)	M(2)—O(81)	2.194 (3)
M(4)—O(40)	2.167 (2)	M(3)—O(61)	2.230 (2)
M(1)—O(11)	2.152 (2)	M(4)—O(51)	2.222 (3)
M(2)—O(11)	2.188 (2)	M(1)···M(2)	3.368 (2)
M(4)—O(11)	2.120 (2)	M(1)···M(3)	3.212 (2)
M(1)—O(21)	2.191 (2)	M(1)···M(4)	3.214 (1)
M(2)—O(21)	2.157 (2)	M(2)···M(3)	3.221 (2)
M(3)—O(21)	2.106 (2)	M(2)···M(4)	3.224 (2)
M(1)—O(31)	2.120 (2)	M(3)···M(4)	3.354 (2)

† M(x) = Mn(x)/Mg(x).

Table 3. Hydrogen-bonding geometry (Å, °)

D—H···A	H···A	D···A	D—H···A
O(51)—H(51O)···Cl(2)	2.17 (1)	3.121 (3)	168 (4)
O(61)—H(61O)···Cl(1)	2.18 (2)	3.127 (3)	164 (4)
O(71)—H(71O)···Cl(4)	2.15 (1)	3.111 (3)	173 (5)
O(81)—H(81O)···Cl(3)	2.20 (2)	3.144 (3)	164 (5)
O(1E)···Cl(3ᶦ)	−	3.13 (4)	−

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

The structure was solved by Patterson methods (*SHELXS86*; Sheldrick, 1990) and refined by a full-matrix least-squares program (*SHELXL93*; Sheldrick, 1993). The refined occupancy of the Mn cations suggested cation mixing; the final structure solution was then refined on the basis of equally populated Mg/Mn sites. The high value of anisotropic displacement parameters for some atoms of the ethanol molecules indicated possible disorder. As a result of this we tried to solve the structure with these atoms disordered over two sites. As a result of such refinement, we found C(61) and C(81) disordered between two positions. Moreover, it was found that all atoms of the uncoordinated ethanol solvent molecule had site-occupation factors of 0.5; C—C and C—O bond lengths had to be restrained to have reasonable values. All disordered C and O atoms were refined isotropically. All C-bonded H atoms were included at calculated positions and refined using a riding model with isotropic displacement parameters equal to $1.2U_{eq}$ of the respective atom. The H atoms of hydroxyl groups (except those of disordered solvent molecule) were located from a difference Fourier map and their coordinates were refined with O—H distances restrained.

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Silver Acetate–Triphenylphosphine Complexes. Acetatobis(triphenylphosphine)silver(I) and its Sesquihydrate, and Bis[acetato(triphenylphosphine)silver(I)] Hydrate and its Hemihydrate

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Abstract

The Ag atom in both independent molecules of acetatobis(triphenylphosphine)silver(I), $[Ag(C_2H_3O_2)(C_{18}H_{15}P)_2]$, is bonded to the triphenylphospine and chelating acetato ligands in a distorted tetrahedral environment. Acetatobis(triphenylphosphine)silver(I) sesquihydrate, $[Ag(C_2H_3O_2)(C_{18}H_{15}P)_2].1.5H_2O$, also crystallizes as two independent molecules, the Ag atoms of which adopt a similar geometry. One $[Ag(C_2H_3O_2)(C_{18}H_{15}P)_2]$ molecule is linked through a water molecule to another complex molecule, which in turn is linked to the symmetry-related first molecule through the other two water molecules, one of which is disordered, affording a chain structure parallel to the *b* axis. Bis(μ-acetato)-$O,O':O;O:O$-bis[(triphenylphosphine)silver(I)] hydrate, $[Ag_2(C_2H_3O_2)_2(C_{18}H_{15}P)_2].-H_2O$, comprises two $[Ag(C_2H_3O_2)(C_{18}H_{15}P)]$ moieties; the acetato ligand behaves as a bidentate chelating anion in one complex moiety but as a monodentate anion in the other. The two moieties link through the singly bonded ester O atom into a dinuclear entity. Neighboring dinuclear entities are linked by hydrogen bonds involving their doubly-bonded carboxyl ends with the disordered water molecule to form a zigzag chain along the *c* axis. In bis-(μ-acetato-$O:O$)bis[(triphenylphosphine)silver(I)] hemihydrate, $[Ag_2(C_2H_3O_2)_2(C_{18}H_{15}P)_2].0.5H_2O$, the two Ag—$O_{carboxyl}$ distances are long enough for the geome-

try of the Ag atoms to be regarded as being essentially trigonal planar in the two complex units. The dinuclear molecule uses its doubly-bonded O atoms to link with the disordered water molecule to afford a hydrogen-bonded tetranuclear entity.

Comment

Acetatobis(triphenylphosphine)silver(I), (I), has been synthesized by reacting silver ketenide with excess triphenylphosphine in refluxing toluene (Femi-Onadeko, 1980), but it can be conveniently prepared by treating silver acetate with two molar equivalents of triphenylphosphine in boiling toluene. This complex is the silver analog of acetatobis(triphenylphosphine)copper(I) (Drew, Othman, Edwards & Richards, 1975), which exists as a monomeric acetato-chelated tetrahedral compound. Both compound (I) and its sesquihydrate, (II), show similar four-coordinate tetrahedral geometries for their Ag atoms. However, the anhydrous compound (I) is packed (d = 1.392 g cm^{-3}) such that there are no solvent-accessible voids in its unit cell, as calculated by *PLATON* (Spek, 1990). The sesquihydrate, (II), is more loosely packed (d = 1.345 g cm^{-3}) and there are four 72 Å^3 voids in the unit cell.

(I)

.1.5H$_2$O

(II)

(III)