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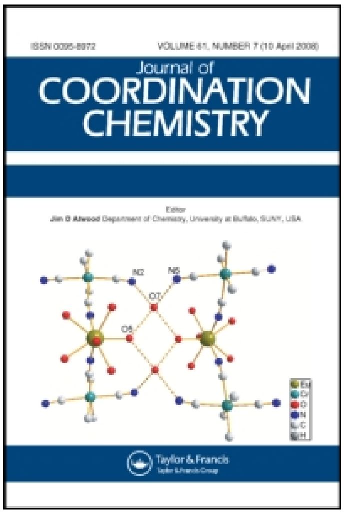

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SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF A METHOXIDEBRIDGED BINUCLEAR MANGANESE(III) COMPLEX WITH BIS(2-OXYPHENYL)DISULFIDE (MP-MP^2); Mn$_2$ (μ-OCH$_3$)$_2$(MP-MP)$_2$(DMSO)$_2$

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SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF A METHOXIDE-BRIDGED BINUCLEAR MANGANESE(III) COMPLEX WITH BIS(2-OXYPHENYL)DISULFIDE (MP-MP^2); Mn$_2$ (μ-OCH$_3$)$_2$(MP-MP)$_2$(DMSO)$_2$

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The title complex $Mn_2(\mu\text{-}OCH_3)_2(MP\text{-}MP)_2(DMSO)_2$, 1, ($MP\text{-}MP^{2-}$ = bis(2-oxyphenyl)disulfide) was obtained by the reaction of $Mn(CH_3CO_2)_2.4H_2O$, H_2MP (*o*-mercaptophenol) and NaOMe in the mixed solvent DMSO/CH_3OH. The structure of 1 was determined by X-ray diffraction methods. Crystal data: $C_{30}H_{34}Mn_2O_8S_6$, triclinic, $P\bar{1}$, a = 8.410(1), b = 8.738(1), c = 12.588 (2)Å, a = 82.85(1), β = 85.78(1), y = 72.38 (1)°, V = 874.1 Å^3, Z = 1, M_r = 824.86, d_x = 1.57 g/cm^3, F(000) = 424, μ (Mo Kα) = 10.8 cm^{-1}, $R(R_w)$ = 0.057(0.061). The molecule consists of two monomers, $Mn(OCH_3)(MP\text{-}MP)(DMSO)$, bridged by the methoxide groups. Each $MP\text{-}MP^{2-}$ ligand provides two phenolate oxygen atoms and one disulfide sulfur atom to the manganese(III) ion in the $Mn_2(\mu\text{-}OMe)_2$ unit. The effective magnetic moment is μ_{eff} = 4.24 μ_B per Mn(III) ion at room temperature.

KEYWORDS: Manganese, methoxide-bridge, *o*-mercaptophenol, disulfide, crystal structure, magnetism

INTRODUCTION

Manganese plays an important role in numerous biological systems such as catalases, ribonucleotide reductase, and the oxygen-evolving complex (OEC) in Photosystem II which all contain active sites of two or more manganese-oxo aggregates.[1-3] These discoveries have stimulated the syntheses and characterization of numerous manganese complexes with oxygen and/or nitrogen donor atoms so as to understand the relationship of structure and function of the active sites.[1-3] Many model complexes with varied nuclearity have appeared in the literature, among which binuclear complexes with bridging oxide and/or carboxylato groups have been studied in some detail,[11-13] yet those with double alkoxide bridges are relatively less investigated.[6-13] Most of the structurally identified binuclear complexes with $Mn(III)_2(\mu\text{-}OR)_2$ unit are manganese Schiff-base complexes[6-12] and only two examples are doubly bridged by methoxide groups.[6-7]

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In our investigation of the coordination chemistry of *o*-mercaptophenol (H_2MP), a series of transition metal complexes with different coordination modes has been prepared.[14–19] In addition, it has been established that H_2MP coordinates oxidatively to manganese(III) to constitute the first Mn(III)-disulfide complex $(Et_4N)[Mn(MP\text{-}MP)_2]$[16] (2) with bis(2-oxyphenyl)disulfide ($MP\text{-}MP^{2-}$. Herein we report a binuclear analogue of 2 with the $Mn_2(\mu\text{-}OCH_3)_2$ unit: $Mn_2(\mu\text{-}OCH_3)_2(MP\text{-}MP)_2(DMSO)_2$ (1).

EXPERIMENTAL

All reagents are commercially available. IR spectra were recorded on a Perkin-Elmer 577 spectrophotometer in KBr pellets. Magnetic susceptibility was measured on a Gouy-Faraday magnetic balance at room temperature *via* the Faraday method with diamagnetic correction.

Preparation of $Mn_2(\mu\text{-}OCH_3)_2(MP\text{-}MP)_2(DMSO)_2$ *(1)*

To a solution of H_2MP (0.63 cm^3, 6 mmole) and $NaOCH_3$ (0.65 g, 12 mmole), obtained by dissolving Na metal in dry MeOH and evaporation of solvent in a mixed solvent consisting of MeOH (20 cm^3) and DMSO (10 cm^3) was added $Mn(CH_3CO_2)_2.4H_2O$(0.75 g, 3 mmole). The reaction mixture was stirred for 30 min and then filtered. The resulting red filtrate was exposed to air for 3 days to give 1 as brown, blocky crystals with a yield of 45%, which were filtered, washed with THF and dried *in vacuo*. *Anal,;* Calcd. for $C_{30}H_{34}Mn_2O_8S_6$: C, 43.68; H, 4.15; Mn, 13.32%. Found: C, 44.25; H, 3.99; Mn, 13.00%. IR(KBr): 288(m), 351(w), 418(m), 452(m), 487(m), 548(s), 608(m), 627(m), 671(w), 682(w), 745(s), 757(s), 851(s), 954(s), 1002(s), 1027(s), 1046(s), 1119(m), 1262(s), 1290(s), 1308(s), 1430(sh), 1454(s), 1579(s) cm^{-1}.

Crystallographic data collection and solution of structure

Crystallographic data for 1 are summarized in Table 1. Diffraction data were collected on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated MoK_a radiation at room temperature. Lp factor, anisotropic decay, and empirical absorption corrections were applied. A total of 1527 unique reflections with $I > 3\sigma(I)$ were used for structure determination. The structure was solved by direct methods and refined by full-matrix least-squares procedures with anisotropic thermal parameters for all the non-hydrogen atoms. Hydrogen atoms were located at idealized positions and added to the structure factor calculations but their positions were not refined. All calculations were performed on a VAX 11/785 computer using the SDP program package.

RESULTS AND DISCUSSION

Compound 1 was prepared by the reaction of $Mn(CH_3CO_2)_2.4H_2O$,H_2MP and $NaOCH_3$ in 1:2:4 ratio in the mixted solvent DMSO + MeOH. In the reaction, both Mn(II) and MP^{2-} were oxidized to Mn(III) and $MP\text{-}MP^{2-}$, respectively. Similar

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Table 1 Crystal Data and Collection Parameters for 1.

Formula	$C_{30}H_{34}Mn_2O_8S_6$
Molecular weight	824.86
Colour	reddish-brown
Crystal system	triclinic
Space group	$P\bar{1}$
a (Å)	8.410(1)
b (Å)	8.738(1)
c (Å)	12.588(2)
α (deg)	82.85(1)
β (deg)	85.78(1)
y (dg)	72.38(1)
V (Å^3)	874.1
Z	1
d_x (g cm^{-3})	1.57
F (000)	424
μ(cm^{-1})	10.8
Crystal dimensions (mm^3)	0.10 × 0.2 × 0.12
Radiation (Å)	Mo Kα (0.71069)
Scan mode	w-2θ
2θ range (deg)	2 to 50.0
No. reflections measured	3219
No. reflections used ($I > 3\sigma(I)$)	1527
Variables	208
R^a	0.057
R_w^b	0.061

$^aR = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$. $^bR_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{½}$ where $w = 1/\sigma^2(F_o^2) + (0.020F_o)^2 + 1.000]$.

oxidative coordination of MP^{2-} to manganese has been reported earlier[16] for the compound $(Et_4N)[Mn(MP\text{-}MP)_2]$ (2), which was the final product of the reaction of MP^{2-} with manganese (II) in CH_3CN or MeOH in the presence of air. In this work, the reaction medium (DMSO/MeOH) promoted the formation of the binuclear complex 1 where the highly polar aprotic molecule DMSO played a major role by occupying one coordination site. The excess base NaOMe easily affords OMe for bridging and completes the octahedral coordination of Mn(III).

Compound 1 crystallizes in the space group $P\bar{1}$ and consists of discrete binucear molecules. The structure of 1 is depicted in Figure 1 and positional parameters, selected atomic distances and bond angles are given in Table 2 and Table 3, respectively.

The dimer 1 consists of two six-coordinate Mn(III) ions which are 3.037 Å apart and bridged by two methoxide groups (O(4) and O(4')) to form an $Mn_2(\mu\text{-}OCH_3)_2$ unit with a crystallographic centre of symmetry. Each Mn(III) is additionally coordinated by two phenolate oxyten atoms and one disulfide sulfur atom from $MP\text{-}MP^{2-}$ and the sixth-coordination site is completed by the oxygen atom of a molecule of DMSO to form a distorted octahedron. The largest deviations of angles around Mn(III) from the thereotical values for an octahedron are found for O(4)-Mn-O(4') (76.7°) and O(1)-Mn-O(4') (165.2°). The Mn(III)-O distances can be classified into three types with the order Mn-O(1,2) (1.865 ~ 1.870 Å) < Mn-O(4,4') (1.925 ~ 1.948 Å) < Mn-O(3) (2.179 Å). The Mn-O(1,2) distances (av. 1.868 Å) are very close to the Mn(III)-$O_{phenoxide}$ bonds in $(Et_4N)[Mn(MP\text{-}MP)_2]$[16] (1.884–1.897 Å) and $[Mn(salen)(AcO)]_n$[20] (salen = N, N'-ethylenebis(salicylideneamide), 1.888

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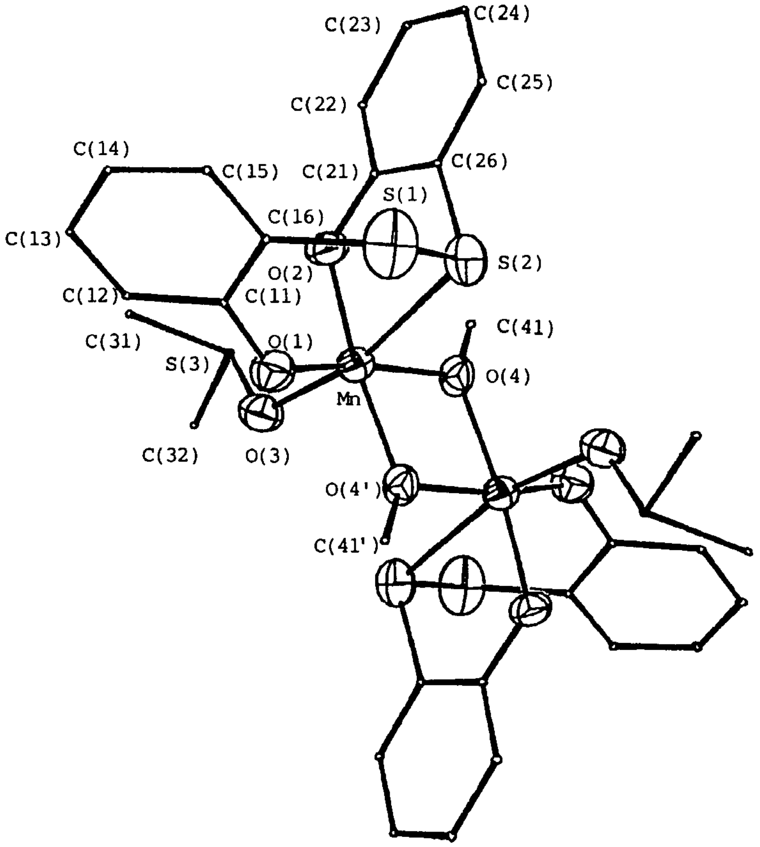

Figure 1 Structure of $Mn_2(\mu\text{-}OCH_3)_2(MP\text{-}MP)_2(DMSO)_2$ showing the atom labelling scheme.

Å) and the Mn(IV)-O bonds (1.874–1.922 Å) in $[Mn(3,5\text{-}(t\text{-}Bu)_2CAT)_3]^{2-}$ (CAT = catecholate)$^{21-22}$ in spite of the different ionic radii of the manganese(IV/III) ions. The bridging Mn-O(4,4') bond distances (av. 1.936 Å) are similar to those corresponding bonds, which are not elongated by Jahn-Teller effect, in the Mn_2O_2 cores (shown in Table 4) of Mn(III) complexes with $Mn_2(OR)_2$ unit such as compounds 3,^{6} 4,^{7} 5,^{13} 6,^{9} 7,^{9}, 8^{10} and 9.^{11} The longest Mn-O(3) bond of 2.176 Å shows weak coordination of the neutral DMSO molecule with Mn(III). The Mn-S(2) distance (2.736 Å) is longer than already reported Mn(III)-thiolate bonds (2.35 Å)$^{14,24-25}$ and is close to the Mn-S bond in the manganese(III) disulfide complex 2^{16} (2.693 Å) and the Mn(II)-disulfide bonds in $Mn(SALPS)CH_3OH.CH_3OH$ (SALPS = *N*, *N'*-[1,1' dithiobis(phenylene)]bis(salicylideneaminato), 2.77 Å) and $[Mn(SALPS)]_2.2CH_3CN$ (2.707, 2.757 Å).^{23} These bond parameters can be reasonably explained by Jahn-Teller elongation along the S(2)-Mn-O(3) axis with the result that the atoms O(1), O(2), O(4) and O(4') occupy the equatorial positions with displacements ranging from -0.0689 to +0.066 Å from the least-squares plane; atoms S(2) and O(3) occupy the apical sites. The other disulfide sulfur atom S(1) is

Table 2 Positional Parameters for 1.

Atom	*x*/*a*	*y*/*b*	*z*/*c*	B(Å^2)
Mn	0.0667(2)	0.4103(2)	-0.0975(1)	2.54(3)
S(1)	0.4416(3)	-0.4897(3)	-0.2492(3)	5.07(7)
S(2)	0.2013(3)	-0.3621(3)	-0.2030(2)	3.88(6)
S(3)	-0.1911(3)	0.2083(3)	-0.1207(2)	3.52(5)
O(1)	0.2754(7)	0.2643(7)	-0.1228(5)	3.5(1)
O(2)	-0.0110(7)	0.4317(6)	-0.2356(4)	3.2(1)
O(3)	-0.0593(7)	0.2267(7)	-0.0516(5)	3.7(1)
O(4)	0.1186(6)	0.4152(6)	0.0481(4)	2.9(1)
C(11)	0.357(1)	0.221(1)	-0.2150(7)	3.3(2)
C(12)	0.377(1)	0.067(1)	-0.2443(7)	3.9(2)
C(13)	0.477(1)	0.013(1)	-0.3334(8)	5.0(3)
C(14)	0.552(1)	0.110(1)	-0.3942(8)	5.1(3)
C(15)	0.531(1)	0.263(1)	-0.3718(8)	4.4(3)
C(16)	0.436(1)	0.321(1)	-0.2805(7)	3.4(2)
C(21)	-0.021(1)	-0.4452(9)	-0.3121(7)	2.8(2)
C(22)	-0.132(1)	-0.420(1)	-0.3934(7)	3.4(2)
C(23)	-0.134(1)	-0.299(1)	-0.4784(8)	4.8(3)
C(24)	-0.030(1)	-0.205(1)	-0.4820(8)	5.0(3)
C(25)	0.075(1)	-0.226(1)	-0.3997(8)	4.6(3)
C(26)	0.079(1)	-0.3451(9)	-0.3148(7)	3.0(2)
C(31)	-0.088(1)	0.053(1)	-0.200(1)	6.4(3)
C(32)	-0.303(1)	0.102(1)	-0.032(1)	6.2(3)
C(41)	0.277(1)	0.352(1)	0.0934(8)	5.0(3)

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Table 3 Selected Bond Distances (Å) and Bond Angles (deg) for 1.

(a) distances			
Mn-S(2)	2.736(3)	Mn-O(1)	1.865(6)
Mn-O(2)	1.870(6)	Mn-o(3)	2.179(6)
Mn-O(4)	1.925(6)	Mn-O(4')	1.948(5)
S(1)-S(2)	2.073(4)	S(1)-C(16)	1.76(1)
S(2)-C(26)	1.770(9)	O(1)-C(11)	1.34(1)
O(2)-C(21)	1.338(9)	S(3)-O(3)	1.515(6)
S(3)-C(31)	1.75(1)	S(3)-C(32)	1.76(1)
O(4)-C(41)	1.41(2)		
(b) angles			
S(2)-Mn-O(1)	85.2(3)	S(2)-Mn-O(2)	77.9(2)
S(2)-Mn-O(3)	166.3(2)	S(2)-Mn-O(4)	101.1(2)
S(2)-Mn-O(4')	88.7(2)	O(1)-Mn-O(2)	96.9(3)
O(1)-Mn-O(3)	95.2(3)	O(1)-Mn-O(4)	91.3(2)
O(1)-Mn-O(4')	165.2(3)	O(2)-Mn-O(3)	88.4(2)
O(2)-Mn-O(4)	171.6(2)	O(2)-Mn-O(4')	94.9(2)
O(3)-Mn-O(4)	92.7(2)	O(3)-Mn-O(4')	93.9(2)
O(4)-Mn-O(4')	76.7(2)	S(2)-S(1)-C(16)	107.6(3)
S(1)-S(2)-C(26)	105.4(4)	Mn-S(2)-S(1)	105.1(1)
Mn-S(2)-C(26)	90.6(4)	Mn-O(1)-C(11)	130.5(5)
Mn-O(2)-C(21)	127.3(5)	Mn-O(3)-S(3)	121.0(3)
Mn-O(4)-Mn'	103.3(2)	Mn-O(4)-C(41)	126.7(5)
Mn'-O(4)-C(41)	126.6(5)		

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Table 4 Important Structural Parameters for the Mn_2O_2 Core in Some Related Binuclear Complexes.

Complex[a]	Mn-Mn (Å)	Mn-O (Å)	O-Mn-O (deg)	ref.
1	3.037	1.925, 1.948	76.7	this work
3	3.144	1.901, 2.227[b]	97.2	6
4	3.192	1.899, 1.910	101.1	7
		2.209[b], 2.220[b]	101.4	
5	3.247	1.911, 2.320[b]	80.22	13
6	3.011	1.926, 1.955	101.8	8
7	3.111	1.900, 2.230[b]	82.9	9
8	2.980	1.963, 1.935	99.5	10
		1.926, 1.942	101.0	
9	3.006	1.941, 1.945	101.6	11
		1.939, 1.939	101.4	

[a] 3: Mn_2 $(SAH)_2$ $(OMe)_2$, SAH = salicyladehydeanthraniloylhydrazonato dianion; 4: [Mn (3,5-diCl-SALPH) $(OMe)]_2$, $H_2$3, 5-diCl-SALPH = 1, 3-bis ((3, 5-dichlorosalicylidene) iminato) propane; 5: $[Mn_2 (SAL)_2(py)_2]^{2-}$ H_2SAL = salicyclic acid; 6: [MnCl (SALAHP) $(MeOH)]_2$, $H_2SALAHP$ = 1-(salicylideneamino)-3-hydroxypropane; 7: $Mn_2(SALMP)_2.2MeCN$, SALMP = 2-(bis(salicylideneamino) methyl) phenolate; 8: $[Mn_2(SALP)(OMe)(NCO)_2(H_2O)_2]$, H_3SALP = 1,5-bis (salicylideneamino)-3-pentanol; 9: $[Mn_2(SALP)(OMe)Cl_2(MeOH)_2]$. [b] Mn-O bonds along the Jahn-Teller axis.

3.837 Å away from the Mn atom and 2.073 Å away fromS(2) with an obvious single S(1)-S(2) bond. The latter distance is typical for ligated disulfide S-S bonds.[23]

Since the dimer lies on the crystallographic inversion centre, the bridging $Mn_2(\mu\text{-}O)_2$ core is perfectly planar. The carbon atoms of the methoxide groups are displaced on opposite sides of the bridging plane by 0.387 Å. Table 4 shows important structural parameters for complexes with an $Mn(III)_2(OR)_2$ unit. The asymmetry of the Mn_2O_2 core can be found in 3, 4, 5 and 7 due to Jahn-Teller elongation along one edge of the Mn_2O_2 core resulting in different lengths of the Mn-O bonds, while the Mn_2O_2 cores in 1, 6, 8 and 9 are symmetrical, with nearly equivalent Mn-O bonds (where the Jahn-Teller elongated axis is perpendicular to the Mn_2O_2 plane). The Mn-Mn distances are in the range 2.980-3.247 Å for all these complexes, indicating no direct metal-metal bonding.

Unlike those structurally investigated complexes[14-19] with *o*-mercaptophenol ligands including 2, the five-membered chelate ring MnS(2)C(26)C(21)O(2) (plane 1) in 1 is not planar, with maximum deviations of atoms -0.4674 to + 0.3239 Å from the least squares plane. Planes MnS(2)S(1)O(1) (plane 2) and O(1)C(11)C(16)S(1) (plane 3) composing the six-membered chelate ring are nearly planar individually (deviations: -0.0693 ~ + 0.0665 Å; -0.0334 ~ + 0.0784 Å, respectively) and form a dihedral angle of 57.3° (planes 2/3). The three planes of the two chelate rings are folded in a "boat" form with a dihedral angle of 48.8° for plane 1 and plane 2. The two phenyl rings form a dihedral angle of 108°.There is no structural data for free HMP-MPH to compare with. Fortunately, we have determined the crystal structure of an analogous ligand, bis (3-hydroxyl-2-pyridyl)disulfide (HMPP-MPPH),[26] in which the two phenyl rings are perpendicular to each other.

The low solubility of 1 in organic solvents such as MeOH, MeCN, DMF, DMSO, Me_2CO, THF, CCl_4, $CHCl_3$ and CH_2Cl_2 forbids the investigation of its solution properties. The measured effective magnetic-moment, μ_{eff}, per manganese(III) ion in the solid state at room temperature for 1 is 4.24 μ_B, which is slightly lower than the d^4 spin-only value of 4.89 μ_B.

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Supplementary material

Complete lists of thermal parameters, bond distances, bond angles, hydrogen positions and observed and calculated structure factors for 1 (7 pages) are available from the authors upon request.

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