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# Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry

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Synthesis, Crystal Structure, and Electrochemical Properties of Two New Manganese Complexes With a Ligand Derived From 1,2-Propanediamine and 2-Hydroxy-5-Methoxybenzaldehyde

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### Synthesis, Crystal Structure, and Electrochemical Properties of Two New Manganese Complexes With a Ligand Derived From 1,2-Propanediamine and 2-Hydroxy-5-Methoxybenzaldehyde

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Two new homo binuclear manganese(III) complexes, [Mn  $(L)(H_2O)]_2(PF_6)_2$ , where  $[H_2L = N, N'-Bis(2-hydroxy-5-methox$ ybenzylidene)-propane-1,2-diamine] have been prepared by reaction of N,N'-bis(2-hydroxy-5-methoxybenzylidene)propane-1,2-diamine as a Schiff-base ligand, manganese(II) acetate and ammonium hexafluorophosphate. The complexes were characterized by spectral, structural and electrochemical studies. X-ray structural analysis shows the presence of dimanganese core in the complexes and the binding of the ligands to the manganese(III) is through N<sub>2</sub>O<sub>2</sub>. The dimeric unit is formed by a double Mn–O bridge of one molecule with its centrosymmetric homologue. The crystal structure of the complexes indicates that in the solid state the complex adopts an octahedral environment of the imine N and hydroxo O with the one axial H<sub>2</sub>O ligand around manganese. There are two coordination/chelation environments present around two manganese centers of each binuclear complex. The electronic spectra of the complex show red shift in the d-d transition. The electrochemical reduction of these complex at a glassy carbon electrode in acetonitrile solution indicates that the first reduction process corresponding to Mn(III)-Mn(II) is electrochemically quasireversible in the range of 0 to -1.0 V.

Keywords binuclear complexes, crystal structure, electrochemical study, manganese(III), homo-dinuclear complexes, N<sub>2</sub>O<sub>2</sub> donors thermal study, UV–vis spectroscopy

### INTRODUCTION

Multidentate Schiff-base ligands played vital role as chelating in the transition metal coordination chemistry. Transition metal complexes of multidentate Schiff-base ligands find applications as model analogues of certain metal enzymes, modifier for selective electrodes, applications in material chemistry, and

catalyst in oxidative addition reactions.<sup>[1-6]</sup> A bimetallic core is used as an active site for metalloenzymes in biological systems. Bimetallic Schiff-base complexes of different structural types have new applications and biological activities.<sup>[7–10]</sup> Design and synthesis of multidentate Schiff-base ligands and their dinuclear complexes have been an interesting area of research because of their importance in basic and applied chemistry. Particular interest has developed in dinuclear complexes as models for metalloproteins.<sup>[11,12]</sup> Schiff-base complexes offer numerous attractive properties, such as electron transport, high thermal stability, and light emission. Organic light emitting diodes have received enormous attention and become an active field of research because of their potential applications in flat panel displays.<sup>[13-19]</sup> The multidenate binucleating Schiff-bases can take up two same or different metal ions. Multidentate binucleating Schiff-bases are reported to form binuclear transition metal complexes.<sup>[20-28]</sup> There are only a limited number of dinuclear manganese complexes.<sup>[29,30]</sup> Differential thermal gravimetry (DTG) and thermogravimetry (TG) are useful to study the modes of thermal decompositions as well as the composition of some metal complexes of Schiff-bases.<sup>[31]</sup> Several researches have proposed that the redox potential in Schiff-base complexes is directly related to chemical characteristics of the entire complex. Thus, there has been a strong interest in determining thermodynamically meaningful redox potentials of manganese Schiff-base complexes and in understanding the relationship between these potentials and the detailed structure of the Schiff-base ligand.<sup>[32-34]</sup> We have focused part of our research program on design of new transition metal complexes,<sup>[35–38]</sup> which constitute one of the more versatile systems in order to mimic biological and industrial catalyses.

In the present research article, we wish to report the structural and electrochemical investigations of two new tetradentate binuclear Schiff-base complex of manganese. The ligand was prepared by the condensation of 2-hydroxy-5-methoxybenzaldehyde, as starting material, with propane-1,2-diamine to afford the corresponding Schiff-base,  $H_2L$ , ligand. The newly prepared binuclear manganese complexes were identified by different physicochemical and spectroscopic

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techniques. The  $H_2L$  ligand acts as  $N_2O_2$  tetradentate sites and can coordinate with two metal ions to form binuclear complexes.

### **EXPERIMENTAL**

### **Materials and Methods**

All solvents and chemicals (Sigma-Aldrich, Germany) were used as received, except for the amines which were distilled under reduced pressure prior to use. UV-vis spectra were recorded on a Varian Cary 500 Scan spectrophotometer (Germany). Infrared spectra (KBr pellets) were obtained on a Bruker FT-IR (Tensor 27) spectrophotometer. Cyclic voltammograms (CV) were recorded by using a SAMA Research Analyzer M-500 (Isfahan, Iran). Three electrodes were utilized in this system, a glassy carbon working electrode, a platinum disk auxiliary electrode, and Ag/Ag+ as reference electrode. The glassy carbon working electrode (Metrohm 6.1204.110, Germany) with 2.0  $\pm$  0.1 mm diameter was manually cleaned with 1  $\mu$ m alumina polish prior to each scan. Tetrabutylammonium tetrafluoroborate (n-Bu)<sub>4</sub>NBF<sub>4</sub>, was used as supporting electrolyte. Acetonitrile was dried over CaH<sub>2</sub>. The solutions were deoxygenated by purging with Ar for 5 min. All electrochemical potentials were calibrated versus internal  $Fc^{+/o}$  ( $E_0 = 0.40$  V versus SCE) couple under the same conditions.

### Synthesis

### *N,N'-Bis*(2-hydroxy-5-methoxybenzylidene)-propane-1,2diamine, ligand

*N*,*N*'-Bis(2-hydroxy-5-methoxybenzylidene)-propane-1,2diamine was prepared by the condensation of 1,2-propanediamine with 2-hydroxy-5-methoxybenzaldehyde (1:2 mol stoichiometric ratio) in MeOH at room temperature and was recrystallized from methanl. Yield: 73%, m.p: 135°C, IR (KBr cm<sup>-1</sup>): 1627  $\upsilon$ (C=N), 2865  $\upsilon$  (C–H sp<sup>3</sup>). UV-vis (Methanol :  $\lambda_{max}$ , nm): 260, 335.

### $[Mn(L)(H_2O)]_2(PF_6)_2$ .TZ, TZ=(5-phenyl-1,3,5-triazine-2,6diamine) (1)

To a stirring solution of Mn(CH<sub>3</sub>COO)<sub>2</sub>. 2H<sub>2</sub>O (0.0662 g, 0.500 mmol) in ethanol (25 mL) was added an equimolar of *N*,*N*<sup>7</sup>-Bis(2-hydroxy-5-methoxybenzylidene)-propane-1,2-diamine (0.5 mmol) and 2 mmol of 5-phenyl-1,3,5-triazine-2,6-diamine and the mixture was stirred for 3 h (Figure 1). The pink solution turned dark brown immediately upon the formation of Mn(III) complex and then 0.500 mmol of NH<sub>4</sub>PF<sub>6</sub> was added. A dark brown microcrystalline solid was produced by slow evaporation of ethanol at room temperature. The product was then recrystallized from methanol-propanol (2:1 v/v) and dark brown crystals suitable for X-ray crystallography were obtained. Yield: 80%, m.p = 269°C. IR (KBr cm<sup>-1</sup>): 850 (PF<sub>4</sub><sup>-1</sup>), 1610, 1550 (C=N), 2900 (C-H sp<sup>3</sup>), 3450, 3500 (NH<sub>2</sub>). UV–vis (Ethanol:  $\lambda_{max}$ , nm): 238, 345.



FIG. 1. Chemical formula of Mn(III) complex, [Mn(L)(H<sub>2</sub>O)]<sub>2</sub>(PF<sub>6</sub>)<sub>2</sub>.TZ (1).

### $[Mn(L)(H_2O)]_2(PF_6)_2$ (2), complex

To a stirring solution of Mn(CH<sub>3</sub>COO)<sub>2</sub>. 2H<sub>2</sub>O (0.0662 g, 0.500 mmol) in methanol (25 mL) was added an equimolar of *N*,*N'*-bis(2-hydroxy-5-methoxybenzylidene)-propane-1,2-diamine (0.500 mmol), and the reaction mixture was stirred for 2 h. To this solution was added 0.500 mmol of NH<sub>4</sub>PF<sub>6</sub>. The resulting dark brown solution was then stirred for 5 min (Figure 2). A dark brown microcrystalline solid was produced by slow evaporation of methanol at room temperature. The product was then recrystallized from methanol-propanol (2:1v/v) and dark brown crystals suitable for X-ray crystallography were obtained. Yield: 70%, m.p = 264°C. IR (KBr cm<sup>-1</sup>): 840 (PF<sub>6</sub><sup>-</sup>), 1609 (C=N), 2900 (C-H sp<sup>3</sup>). UV–vis (Methanol,  $\lambda_{max}$ , nm): 237, 360.

## X-Ray crystallography for $[Mn(L)(H_2O)]_2(PF_6)_2$ .TZ (1) and $[Mn(L)(H_2O)]_2(PF_6)_2$ (2)

Dark brown crystal of complex 1 and 2 was mounted with a cryoloop and flash-cooled by cold nitrogen stream. All measurements were made at 193(2) K on a Rigaku RAXIS RAPID imaging plate area detector (Japan) with graphite



FIG. 2. Chemical formula of Mn(III) complex,  $[Mn(L)(H_2O)]_2(PF_6)_2$  (2).

$[Mn(L)(H_2O)]_2(PF_6)_2.TZ (1)$		(2)	
Chemical formula (moiety) Chemical formula (total) Formula weight Temperature Radiation, wavelength Crystal system, space group Unit cell parameters Cell volume Z Calculated density Absorption coefficient $\mu$ F(000) Crystal color and size Reflections for cell refinement Data collection method	$\begin{array}{c} C_{28}H_{35}F_{6}MnN_{7}O_{8}P\\ C_{28}H_{35}F_{6}MnN_{7}O_{8}P\\ 797.54\\ 193(2) K\\ MoK\alpha, 0.71075 Å\\ monoclinic, P2_{1}/n\\ a = 10.6103(17) Å \alpha = 90^{\circ}\\ b = 20.372(4) Å \beta = 105.349(4)^{\circ}\\ c = 16.087(3) Å \gamma = 90^{\circ}\\ 3353.2(10) Å^{3}\\ 4\\ 1.580 \text{ Mg/ m}^{-3}\\ 0.53 \text{ mm}^{-1}\\ 1640\\ dark \text{ brown, } 0.2 \times 0.2 \times 0.2 \text{ mm}^{3}\\ 28 (\theta \text{ range } 3.09{-}27.48^{\circ})\\ \text{Rigaku RAXIS-RAPID}\\ diffractometer $\omega$ scans \\ \end{array}$	Chemical formula (moiety) Chemical formula (total) Formula weight Temperature Radiation, wavelength Crystal system, space group Unit cell parameters Cell volume Z Calculated density Absorption coefficient $\mu$ F(000) Crystal color and size Reflections for cell refinement Data collection method	C <sub>38</sub> H <sub>44</sub> Mn <sub>2</sub> N <sub>4</sub> O <sub>10</sub> <sup>2+</sup> ·2(F <sub>6</sub> P <sup>1-</sup> )·2(H <sub>2</sub> O) C <sub>38</sub> H <sub>44</sub> Mn <sub>2</sub> N <sub>4</sub> O <sub>10</sub> <sup>2+</sup> ·2(F <sub>6</sub> P <sup>1-</sup> )·2(H <sub>2</sub> O) 1132.62 193(2) K MoKα, 0.71075 Å monoclinic, P2 <sub>1</sub> /n a = 11.9845(7) Å α = 90° b = 13.0588(9) Å β = 107.709(4)° c = 15.8540(10) Å γ = 90° 2363.6(3) Å <sup>3</sup> 2 1.620 Mg/m <sup>-3</sup> 0.71 mm <sup>-1</sup> 1176 Block, brown, 0.3 × 0.2 × 0.2 mm <sup>3</sup> 28 (θ range 3.09–27.48°) Rigaku RAXIS-RAPID diffractometer
$\theta$ range for data collection Index ranges Completeness to $\theta = 24.0^{\circ}$ Reflections collected Independent reflections Reflections with $F^2 > 2\sigma$ Absorption correction Min. and max. transmission Structure solution Refinement method Weighting parameters a, b Data/restraints/parameters Final R indices [ $F^2 > 2\sigma$ ] R indices (all data) Goodness-of-fit on F <sup>2</sup> Extinction coefficient Largest and mean shift/su	3.09–27.48.0° h –17 to 16, k –17 to 17, l –18 to 17 99.4% 4940 2854 (R <sub>int</sub> = 0.042) 2015 multi-scan Higashi.t(1995) 0.892 and 0.892 direct methods Full-matrix least-squares on $F^2$ 0.0611, 1.1171 2854/2/237 R1 = 0.0659, wR2 = 0.1450 R1 = 0.1043, wR2 = 0.1605 1.167 0.012(2) 0.000 and 0.000 0.34 and –0.24 e Å <sup>-3</sup>	$\theta$ range for data collection Index ranges Completeness to $\theta = 24.0^{\circ}$ Reflections collected Independent reflections Reflections with $F^2 > 2\sigma$ Absorption correction Min. and max. transmission Structure solution Refinement method Weighting parameters a, b Data/restraints/parameters Final R indices $[F^2 > 2\sigma]$ R indices (all data) Goodness-of-fit on $F^2$ Extinction coefficient Largest and mean shift/su Largest diff. peak and hole	ω scans 3.09–27.48.0° h –17 to 16, k –17 to 17, 1–18 to 17 99.4% 4940 2854 (R <sub>int</sub> = 0.042) 2015 multi-scan Higashi.t(1995) 0.892 and 0.892 direct methods Full-matrix least-squares on $F^2$ 0.0611, 1.1171 2854/2/237 R1 = 0.0659, wR2 = 0.1450 R1 = 0.1043, wR2 = 0.1605 1.167 0.012(2) 0.000 and 0.000 0.34 and -0.24 e Å <sup>-3</sup>

TABLE 1 Crystal data and structure refinement for  $[Mn(L)(H_2O)]_2(PE_c)_2$  TZ (1)

TABLE 2 Crystal data and structure refinement for  $[Mn(L)(H_2O)]_2(PF_6)_2$ 

monochromated Mo-K $\alpha$  radiation. Absorption corrections were applied by the numerical method.<sup>[39]</sup> Crystal data, together with other relevant information on structure determination, are listed in Tables 1 and 2. The structure was solved by the direct method using SIR2004<sup>[40]</sup> and refined on  $F^2$  with all independent reflections by full-matrix least-square method using SHELXL97 program.<sup>[41]</sup> The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were introduced at the positions calculated theoretically and treated with riding models.

### Electrochemical studies

The CV of complex was conducted at  $25^{\circ}$ C under an argon atmosphere using acetonitrile solutions containing 0.05 mol dm<sup>-3</sup>

 $(n-Bu)_4NBF_4$  as supporting electrolyte and complex concentrations of about  $3 \times 10^{-3}$  mol dm<sup>-3</sup>. The ligands are electroinactive over a range of +2.0 to -2.0 V.

### **RESULTS AND DISCUSSION**

### Synthesis

Two Mn(III) complexes have been prepared by the reaction of an equimolar of N,N'-Bis(2-hydroxy-5-methoxybenzylidene)propane-1,2-diamine and manganese(II) acetate in the presence of the appropriate axial ligand (water) in aerobic conditions (Figures 1 and 2). The air oxidation was continued for a period of 3 h, which the dark brown color solution resulted. Dark



FIG. 3. ORTEP drawing of [Mn(L)(H<sub>2</sub>O)]<sub>2</sub>(PF<sub>6</sub>)<sub>2</sub>.TZ, TZ=(5-phenyl-1,3,5-triazine-2,6-diamine) (1) with the atom numbering scheme. The displacement ellipsoids are drawn at the 50% probability level (color figure available online).

brown crystals of these complexes were obtained in good yield (70-80%).

### **Structural Studies**

X-ray structural analysis of 1 and 2 are shown in Figures 3 and 4, respectively. X-ray structural analysis shows the presence of dimanganese core in the complexes and the binding of the ligands to the manganese(III) is through  $N_2O_2$ . The equatorial plane of Mn<sup>III</sup> has two nitrogen and two oxygen atoms of the  $N_2O_2$  ligand with the bond distances of Mn1–O1 1.84(9), Mn1–O3 1.91(2), Mn1–N1 1.99(1), and Mn1–N2 1.97(1) Å. The dimeric unit is formed by a double Mn-O bridge of one molecule with its centrosymmetric homologue. The crystal structures of the complexes indicate that in the solid state



FIG. 4. ORTEP drawing of [Mn(L)(H<sub>2</sub>O)]<sub>2</sub>(PF<sub>6</sub>)<sub>2</sub> (2) with the atom numbering scheme. The displacement ellipsoids are drawn at the 50% probability level (color figure available online).



FIG. 5. Cyclic voltammogram of  $[Mn(L)(H_2O)]_2(PF_6)_2$  in acetonitrile solution stored for 48 h at 293 K. Scan rate: 100 mV/s.  $c = 3.0 \times 10^{-3}$ .

the complex adopts an octahedral environment of the imine N and hydroxo O with the one axial H<sub>2</sub>O ligand around manganese. There are two coordination/chelation environments present around two manganese centers of each binuclear complex. One axial position around each Mn is occupied by one oxygen atom and another axial position by water. Manganese oxygen bond distances in axial positions, Mn1–O5 2.21(9), Mn1–O3(axi) 2.42(1) Å, are significantly longer than those in the equatorial planes, consistent with the Jahn–Teller elongation of high-spin d<sup>4</sup> Mn<sup>III</sup> in the axial direction and the difference in ligand type. For complex **1** the bond distances of N2O2 in equatorial positions are as followings: Mn1–O1 1.85(2), Mn1–O2 1.89(6), Mn1–N1 1.98(3), and Mn1–N2 1.97(6) Å.

### **Voltammetric Characterization**

The CV of complex was measured in acetonitrile solution. The CV exhibits irreversible mechanism (Figure 5). In cathodic and anodic voltamogram, two peaks were observed which indicates of reduction of Mn(III) to Mn(II) and oxidation of Mn(II) to Mn(III). These results demonstrate that complex is dimmer in solution phase and consistent with dimmer solid state structure.

### **Spectroscopic Properties**

The electronic spectra of complexes 1 and 2 have maxima at 238 and 345 nm for 1 and 237 and 360 nm for 2. The absorptions at 232 and 238 nm can be related to the spin allowed  $\pi -\pi^*$  azomethane intraligand transition.<sup>[42]</sup> The band at 360 nm in the UV region can be assigned to spin-allowed metal-toligand charge transfer transition.<sup>[43–45]</sup> FT-IR spectral data of the complexes were compared with those of the uncomplexed ligand. *N*,*N'*-bis(2-hydroxy-5-methoxybenzylidene)propane-1,2diamine ligand show a broad band characteristic of the OH group in the 3300–3500 cm<sup>-1</sup> region. The disappearance of this band in the FT-IR spectra of the complexes is indicative of the fact that the tetradentate  $N_2O_2$  ligands are coordinated as dianions. The (C=N) band appearing at 1620 cm<sup>-1</sup> in the ligands is shifted to lower frequencies by 13–25 cm<sup>-1</sup> in the corresponding Mn complexes, indicating that the ligands are coordinated to the metal ions through the imino groups.

### CONCLUSION

We have prepared two new homo binuclear manganese(III) complexes. X-ray structural analysis of complexes showed the presence of dimanganese core in the complexes and the binding of the ligands to the manganese(III) through  $N_2O_2$ . The dimeric unit is formed by a double Mn–O bridge of one molecule. The CV of complex demonstrate that complex present only a single species in acetonitrile solution and the redox processes may be described as Mn(III)/Mn(II) and Mn(II)/Mn(III).

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