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Group VI Dinuclear Oxo Metal Complexes of Salicylideneimine-2-anisole Schiff Base

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ABSTRACT

Interaction of the Schiff base salicylideneimine-2-anisole (salanH) with $\text{Cr}(\text{CO})_6$ yielded the dicarbonyl derivative $\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$. The dinuclear oxo complex $\text{M}_2\text{O}_4(\text{salan})_2$, $\text{M} = \text{Mo}$ and W , was isolated from the reaction of $\text{M}(\text{CO})_6$ with salanH. Elemental, spectroscopic and magnetic studies of the reported complexes allowed structures to be proposed. The thermal properties of the complexes were investigated by thermogravimetry.

Key Words: Schiff bases; Chromium; Metal carbonyls; Complexes; IR spectra; Thermal analysis.

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INTRODUCTION

Various transition and inner-transition metal complexes with bi-, tri- and tetradentate Schiff bases containing nitrogen and oxygen donor atoms play important roles in biological systems, and represent interesting models for metalloenzymes that efficiently catalyze the reduction of dinitrogen and dioxygen.^[1,2] Furthermore, complexes of chromium, manganese, nickel and ruthenium with a wide variety of Schiff bases, which have donor atoms such as N_2O_2 and N_4 around the metal ion, have been used as catalysts for epoxidation reactions.^[3] These complexes bind reversibly to molecular oxygen with a change in oxidation state of the metal.^[4-7] On the other hand, the chemistry of oxo metal complexes with Schiff bases is well established. For example, dioxomolybdenum(VI) and dioxotungsten(VI) complexes of two isomeric Schiff bases, derived from salicylaldehyde and 2-hydroxy-1-naphthaldehyde, have been isolated and characterized.^[8] Also, the reactions of $M(CO)_6$, $M=Cr$ and Mo , with bis(2-hydroxyacetophenone)ethylenediimine (hapenH₂) under atmospheric pressure give the oxo derivatives $M(O)(hapen)$ with the metal atom in the + 4 formal oxidation state.^[9] Furthermore, interaction of the Schiff base 2-hydroxyacetophenonepropylimine (happramH) with $M(CO)_6$, $M=Cr$ or Mo , under reduced pressure, gave the dicarbonyl complex $M(CO)_2(happramH)_2$. On the other hand, the complex $MoO(happram)_2$ was isolated from the reaction of $Mo(CO)_6$ with happramH in air.^[10] Interest in studies of oxomolybdenum and oxotungsten complexes stem from their useful application in heterogeneous catalysis, especially for epoxidation of olefins.^[11]

In this paper, the thermal reactions of $M(CO)_6$, $M=Cr$, Mo , and W with salicylideneimine-2-anisole (salanH) (Figure 1) are described.

EXPERIMENTAL

The compounds $M(CO)_6$, where $M=Cr$, Mo and W , were purchased from Aldrich. Salicylideneimine-2-anisole was prepared as described in the

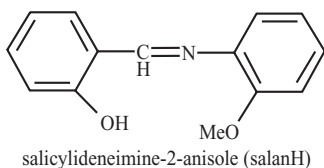


Figure 1.

literature.^[12] All the solvents were of analytical reagent grade and they were purified by standard methods.

Infrared measurements (KBr pellets) were carried out on a Shimadzu 8000 FT-IR spectrometer. Electronic absorption spectra were measured on a Unicam UV2-300 UV-vis spectrophotometer. Samples of concentrations *ca.* 1×10^{-5} M in DMSO, DMF, THF and CH_2Cl_2 were measured against the solvent in the reference cell. NMR measurements were performed on a Varian-Mercury 300 MHz spectrometer. Samples were dissolved in $(\text{CD}_3)_2\text{SO}$ with TMS as the internal reference. Magnetic susceptibility (Gouy method) and ESR measurements of the complexes were carried out on a Sherwood Scientific magnetic susceptibility balance, and a Bruker ESR spectrometer model EMX. Thermogravimetric (TG) analysis was carried out, under N_2 atmosphere, with a heating rate of $10^\circ\text{C}/\text{min}$ using a Shimadzu DT-50 thermal analyzer. Also, the complexes were characterized by elemental analysis (Perkin-Elmer 2400 CHN elemental analyzer) and mass spectroscopy (Finnigan MAT SSQ 7000). Table 1 gives the elemental analysis and mass spectrometry data for the complexes.

Synthesis of $\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$ Complex

A mixture of $\text{Cr}(\text{CO})_6$ (0.12 g; 0.54 mmol) and salicylideneimine-2-anisole (0.13 g; 0.55 mmol) in about 30 ml THF was heated to reflux under atmospheric pressure for 12 h. The color of the reaction mixture changed from yellow to light brown and finally to green with precipitation of a deep green solid. The reaction mixture was cooled and the residue was isolated by filtration. The complex was washed several times with boiling petroleum ether and then recrystallized from DMF. The complex was left to dry *in vacuo* for several hours (yield 70%).

Table 1. Elemental analysis and mass spectrometry data of the complexes.

Complex	Found (calcd.) (%)			Mass spectrometry	
	C	H	N	Mol. wt.	m/z
$\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$	55.6 (55.9)	3.7 (3.8)	4.4 (4.3)	644.53	617 [P-CO] ⁺
$\text{Mo}_2\text{O}_4(\text{salan})_2$	46.9 (47.5)	3.2 (3.4)	3.9 (4.0)	708.48	709 [P] ⁺
$\text{W}_2\text{O}_4(\text{salan})_2$	38.2 (38.0)	2.6 (2.7)	3.1 (3.2)	884.18	869 [P-CH ₄] ⁺



Syntheses of $\text{Mo}_2\text{O}_4(\text{salan})_2$ and $\text{W}_2\text{O}_4(\text{salan})_2$ Complexes

A similar procedure was employed as for the $\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$ complex, but using different reaction periods.

RESULTS AND DISCUSSION

Thermal reaction of salicylideneimine-2-anisole (salanH) with $\text{Cr}(\text{CO})_6$ resulted in the formation of a dinuclear complex with a molecular formula $\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$. The IR spectrum of the salanH ligand displayed bands due to $\nu(\text{OH})$, $\nu(\text{C}=\text{N})$, $\nu(\text{CO})$ due to phenolic and methoxy groups, Table 2. The IR spectrum of the chromium complex showed that the bands exerted appropriate shifts relative to those of the ligand itself, Table 2. The $\nu(\text{C}=\text{N})$ and the $\nu(\text{C}-\text{O})$ methoxy bands of the salanH ligand moiety in the complex was shifted to lower frequency, while the $\nu(\text{C}-\text{O})$ phenolic band was displaced to higher frequency (Table 2). Also, the spectrum showed that the $\nu(\text{OH})$ band had disappeared. These observations were consistent with coordination of chromium to the azomethine nitrogen atom and to the two oxygen atoms of the phenolic and methoxy groups.^[10,13,14] The removal of the hydrogen atom of the OH group upon complexation indicated that ligand was bound to the metal oxidatively. On the other hand, the IR spectrum of $\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$ complex displayed two strong bands at 1944 and 1852 cm^{-1} due to symmetric and asymmetric stretching frequencies of two CO groups.^[15] Furthermore, the IR spectrum of the complex displayed two medium bands, which could be assigned as Cr-O-Cr stretching frequencies.^[15] Therefore, according to the spectroscopic observations, it was expected that each chromium would have a +3 formal oxidation state. Measurements of the magnetic susceptibility showed that the complex has an effective magnetic moment of 3.07 BM. This value is lower than the spin only value of three unpaired electrons for each chromium. This may indicate the presence of an intramolecular antiferromagnetic interaction between the two Cr(III) centers.^[16,17] The ESR spectrum of a powdered sample of $\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$, measured at room temperature, exhibited a broad isotropic single line with a $\langle g \rangle$ value of 2.161.^[16] Isotropic lines of this type are usually observed, either due to intramolecular spin exchange leading to broadening of the line, or due to the occupancy of the unpaired electrons in degenerate orbitals.^[18] The nature and pattern of such an ESR spectrum suggested an almost octahedral environment around each chromium(III) in the complex.^[16] Thus, the structure of the complex may have each chromium in an octahedral arrangement with a ligand bound



Table 2. Important IR data for salicylideneimine-2-anisole and its chromium, molybdenum and tungsten complexes.

Compound	IR data (cm^{-1}) ^a						
	$\nu(\text{O-H})$	$\nu(\text{C}=\text{N})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{O}-\text{M})$	$\nu(\text{C}=\text{O})$	$\nu(\text{C}-\text{OH})$	$\nu(\text{C}-\text{OCH}_3)$
SalanH	3433 (b)	1612 (s)	—	—	—	1242(m)	1149 (m)
$\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$	—	1597 (s)	—	610 (m)	1944 (s)	1257 (s)	1111 (m)
				532 (m)	1852 (s)		
$\text{Mo}_2\text{O}_4(\text{salan})_2$	—	1605 (s)	956 (s)	630 (m)	—	1265(m)	1130 (m)
			900 (s)	571 (m)			
$\text{W}_2\text{O}_4(\text{salan})_2$	—	1620 (s)	980 (m)	632 (m)	—	1265(m)	1150 (m)
			810 (vs)	586 (m)			

^as, strong; m, medium; b, broad.

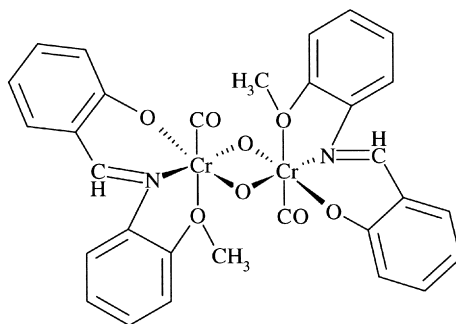
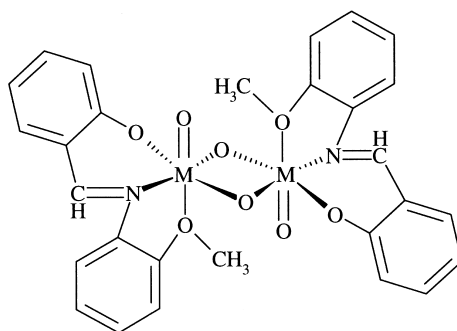


Figure 2.

to the metal through the ONO tridentate sites. Two bridged oxygen atoms as well as a terminal carbonyl are also attached to each chromium atom (Figure 2).

Reactions of $M(CO)_6$, $M=Mo$ and W , with $salanH$ in THF gave complexes with a molecular formula $M_2O_4(salan)_2$. The IR spectra of both molybdenum and tungsten complexes showed patterns similar to that of the ligand, with the appropriate shifts, Table 2. Also, they showed the disappearance of the OH bands of ligand moieties. In addition, the spectra displayed asymmetric and symmetric stretching frequencies due to $M=O$ and $M-O-M$ bonds, Table 2. The 1H NMR spectrum of the molybdenum and tungsten complexes exhibited a multiplet and two singlets due to the protons of the phenyl, methyl and $CH=N$ groups. These signals were

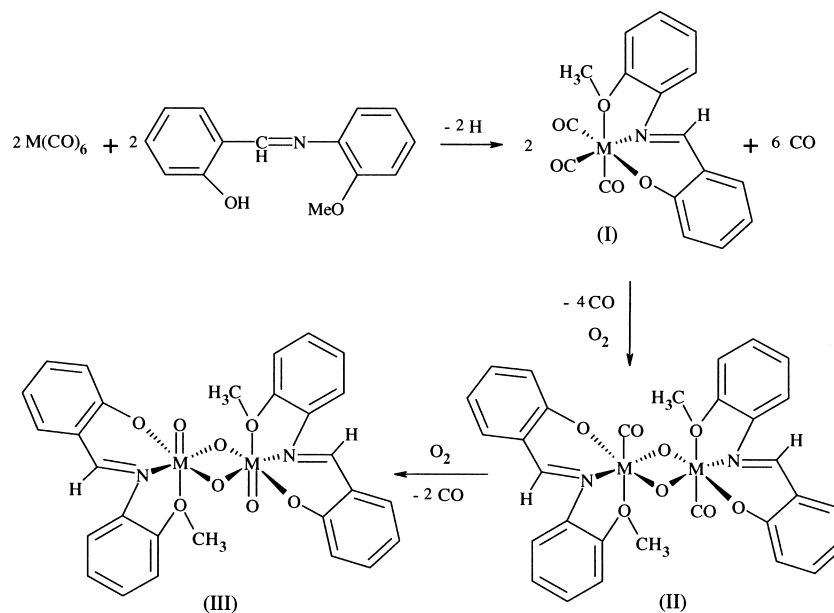


$M = Mo, W$

Figure 3.

broad due to the paramagnetic characteristics of these complexes, *vide infra*. These signals showed downfield shifts relative to those of ligand itself [7.3 (m), 2.55 (s) and 9.01 (s) ppm]. The ^1H NMR spectra of the two complexes also showed the disappearance of the signal due to the hydroxyl proton of the ligand moieties (13.86 ppm). The absence of this signal in the spectra of the complexes indicated the coordination of the phenolic oxygen to the metal through oxidative addition. The ESR spectra of powdered samples of $\text{Mo}_2\text{O}_4(\text{salan})_2$ and $\text{W}_2\text{O}_4(\text{salan})_2$ measured at room temperature exhibited a sharp isotropic single line with a $\langle g \rangle$ value of 1.915 for the molybdenum complex and 2.111 for the tungsten complex.^[16] Isotropic lines of this type are usually observed for a single unpaired electron.^[16] Therefore, it can be concluded that the molybdenum and tungsten existed in a +5 (d^1) formal oxidation state. According to the spectroscopic studies, the complex consisted of two metals coordinated to two bridged oxygen atoms. Each metal also coordinated to a tridentate salan ligand and a terminal oxygen, Figure 3.

A proposed mechanism for the formation of the oxo complexes $\text{M}(\text{O})(\text{hapen})$, $\text{M}=\text{Cr}$ or Mo ; hapenH_2 = the tetradentate bis-(2-hydroxy-acetophenone)ethylenediimine, suggested that it was formed through a



Scheme 1.



six coordinate intermediate $M(\text{CO})_2(\text{happenH}_2)$.^[9] On the other hand, $\text{MoO}(\text{happam})_2$ complex, formed from interaction of the bidentate Schiff base 2-hydroxyacetophenonepropylimine (happamH) with $\text{Mo}(\text{CO})_6$, revealed that it was formed through a dihydride intermediate with one of the hydride ligands trans to a CO group. Trans labilization effect of the hydride ligand underwent loss of that CO group. Reaction with an oxygen molecule gave the oxo derivative $\text{MoO}(\text{happam})_2$.^[10] For the present dinuclear oxo complexes, it can be speculated that interaction of the tridentate Schiff base with the hexacarbonyls of group 6 would lead to formation of the tricarbonyl intermediate I, Scheme 1. Reaction of that intermediate with O_2 might give the dinuclear complex II (the chromium derivative). Further reaction with oxygen would result in the formation of the dinuclear oxo derivative III (the molybdenum and tungsten complexes), Scheme 1. It was previously found that reactions of $M(\text{CO})_6$, $M=\text{Mo}$ and W with the bidentate ligand dimethylglyoxime (DMG) in presence of oxygen yielded the dinuclear oxo complex $M_2\text{O}_6(\text{DMG})_2$ through similar mechanism of formation.^[19,20]

Uv-vis. Studies

The electronic absorption spectra of the salanH Schiff base and its complexes were investigated in different polar solvents (Table 3). Two absorption bands were observed for salanH in DMF, DMSO, THF and

Table 3. The UV-vis. data for the reported complexes in different solvents.

Compound	Solvent			
	DMSO	DMF	THF	CH_2Cl_2
SalanH	294(105300)	291(102717)	288 (9899)	280 (4300)
	345(13030)	348(123140)	349 (7355)	348 (6510)
$\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$	290(26726)	299 (91770)	296 (21010)	302 (6118)
	350 (6440) ^a	350 (22540) ^a	350 (4830) ^a	355 (3220) ^a
	445 (1610) ^b	445 (5990) ^b	445 (1370) ^b	490 (2093) ^b
$\text{Mo}_2\text{O}_4(\text{salan})_2$	288 (65930)	307 (6110)	Insol.	Insol.
	350 (15930) ^a	450 (354) ^b		
	440 (3540) ^b			
$\text{W}_2\text{O}_4(\text{salan})_2$	293 (4138)	301 (3536)	Insol.	Insol.
	360 (420) ^a	370 (1550) ^a		
	450 (220)	450 (332) ^b		

^ashoulder.

^bbroad.

CH_2Cl_2 in the ranges of 280–294 and 345–349 nm due to $\pi\text{-}\pi^*$ and $\text{n-}\pi^*$ transitions, respectively. It was found that the absorbance decreased as the polarity of the solvent decreased, Table 3. In DMSO, on going from ligand to complex, the $\pi\text{-}\pi^*$ electronic transition band at 294 showed a hypsochromic shift, while the $\text{n-}\pi^*$ band at 345 nm showed a bathochromic shift with a considerable change in absorbance. These observations are consistent with complex formation (Table 3). For the complexes MoO_2L and WO_2L , where L=a Schiff base derived from o-aminobenzyl alcohol or hydroxybenzyl amine, similar trends were observed.^[18] In addition, the three complexes exhibited absorption bands at 440–450 nm that could be due to ligand-to-metal charge transfer transitions.^[21,22]

Thermogravimetric Analysis

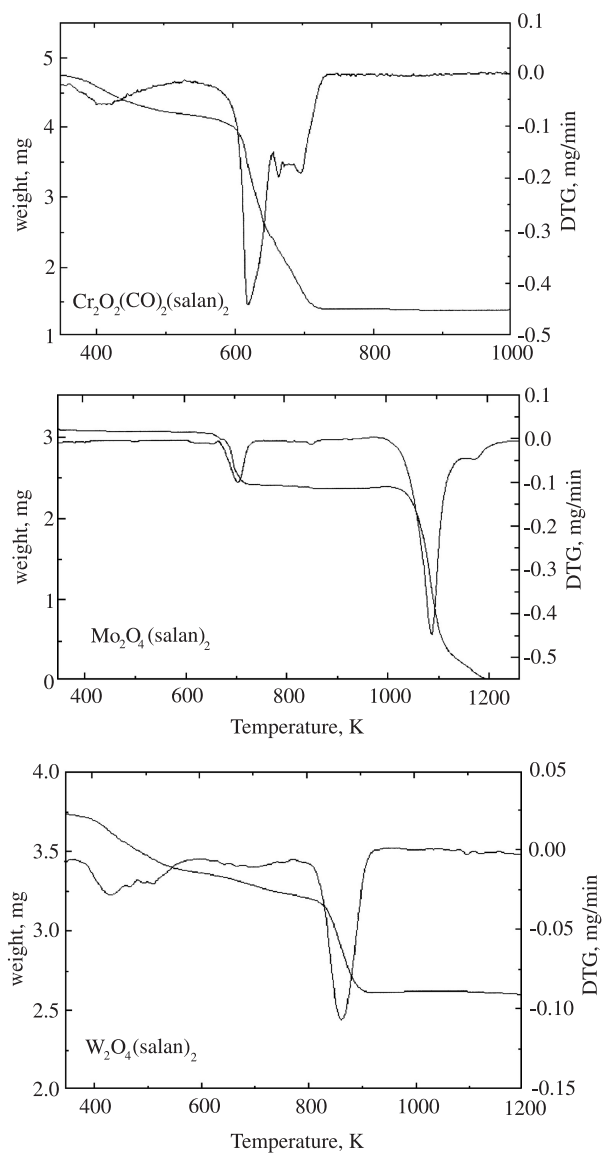
The thermal studies of the chromium, molybdenum and tungsten complexes were carried out using thermogravimetry (TG) and differential thermogravimetry (DTG). Typical TG and DTG plots for the three complexes are given in Figure 4.

The TG plot of $\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$ complex showed that it was decomposed in two steps. The first decomposition step, which occurred in the temperature range 392–502 K, was weak and had a broad peak with a net weight loss of 13.7% (Figure 4). The percentage weight loss was consistent with the elimination of two CO groups and a C_2H_6 species. On the other hand, the second decomposition step was a composite peak with three successive and overlapped decomposition peaks. The total weight loss of the second decomposition step was found to be 63.0% and corresponded to a material decomposition to give finally chromium oxide (Cr_2O_3).

The thermogravimetric studies of the $\text{Mo}_2\text{O}_4(\text{salan})_2$ complex showed that it was decomposed in two well-defined and non-overlapping steps, Figure 4. The successive weight losses were observed in the temperature range 670–1240 K. The first decomposition peak, occurred at 670–740 K with a percentage weight loss of 22.6%, was due to a loss of a C_8H_{14} species. The second step (1050–1240 K) had a net weight loss of 77.4% and corresponded to the decomposition and volatilization of the rest of the compound.

The TG plot of the complex $\text{W}_2\text{O}_4(\text{salan})_2$ showed that it decomposed, like the molybdenum analog, in two steps within the temperature range 387–930 K. The percentage weight loss in the first decomposition peak (9.9%) was attributed to the elimination of a C_7H_8 species. The second decomposition peak, occurred at 774–930 K with a 18.8% weight loss, was due to a material decomposition to leave a metallic residue.



**Figure 4.**

CONCLUSION

The three oxo complexes, $\text{Cr}_2\text{O}_2(\text{CO})_2(\text{salan})_2$, $\text{Mo}_2\text{O}_4(\text{salan})_2$ and $\text{W}_2\text{O}_4(\text{salan})_2$ result from the reactions of $\text{M}(\text{CO})_6$ with the *salanH* Schiff base. The bands in the IR spectra of the complexes revealed appropriate shifts due to complex formation. Magnetic studies showed that the three complexes were paramagnetic. Chromium existed as $\text{Cr}(\text{III})$, d^3 , while both molybdenum and tungsten were present as $\text{M}(\text{V})$, d^1 , species. The thermogravimetric studies of the complexes showed multistep degradation. Although both Mo and W have similar structures, they had different thermal decomposition mechanisms.

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