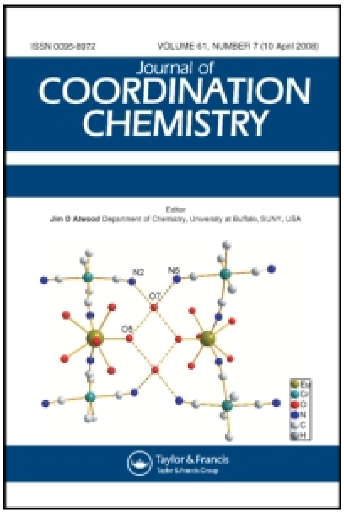

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Omyma A. M. Ali[a]
[a] Chemistry Department, University College for Girls, Ain shams University, Cairo, Egypt

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Synthesis and characterization of molybdenum complexes with salicylideneimine-2-anisole and certain heterocyclic nitrogen ligands

OMYMA A. M. ALI*

Chemistry Department, University College for Girls,
Ain shams University, Cairo, Egypt

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Reactions of $Mo(CO)_6$ with salicylideneimine-2-anisole (Schiff-base, salanH), in the presence of certain heterocyclic nitrogen compounds in THF produced different complexes depending on the reaction conditions. In air, three dinuclear complexes $[O_2(salanH)Mo(\mu\text{-}O)_2MoO_2L]$ were isolated, L = 2,2′-bipyridine (bpy) or 1,10-phenanthroline (phen) or 2,9-dimethy1-4,7-diphenyl-phenanthroline (dim). Under reduced pressure, the corresponding reactions resulted in the formation of the tetracarbonyl derivatives, $[Mo(CO)_4(salanH)](L)$. The complexes were characterized by elemental analyses, spectroscopic and magnetic studies. The thermal properties of the complexes were investigated using thermogravimetry.

Keywords: Molybdenum hexacarbonyl; Heterocyclic nitrogen compounds; Spectra; Schiff-bases; Thermal analysis

1. Introduction

Transition metal complexes with Schiff bases containing nitrogen and oxygen donor atoms play an important role in biological systems [1–3]. Furthermore, the chemistry of molybdenum-oxo-complexes with Schiff-base ligands has received attention because of its relevance to the active sites of molybdoenzymes [4]. It is well known that biological redox processes of molybdoenzymes involve Mo(VI) and Mo(IV) [5]. In addition, extended X-ray absorption fine structure (EXAFS) spectroscopic studies have implicated the presence of oxygen and nitrogen at the active sites of oxo-transfer molybdoenzymes [6, 7]. Three complexes $[M(CO)_4(HDpyF)]$ [M = Cr or W] and $[Mo(CO)_3(HDpyF)(CH_3CN)]$ have been prepared by reaction of *bis*(2-pyridyl)formamidine (HDpyF) with the corresponding metal hexacarbonyl in THF and CH_3CN, respectively [8]. The HDpyF ligand was found to coordinate to metal centres in different bidentate bonding modes. Also, the complexes showed supramolecular structures in the solid state by intermolecular hydrogen bonding. The oxomolybdenum(IV) complexes with ONS donor Schiff-bases (LH) in the form of thiocarbodihydrazone of salicylaldehyde and substituted salicylaldehydes were

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*Email: omaymaahmed92@yahoo.com

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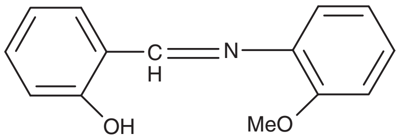
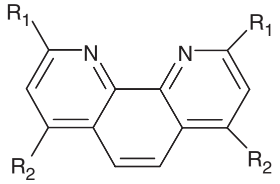


Salicylideneimine-2-anisole; salanH



R_1, R_2=H, 1,10-phenanthroline; phen

$R_1=CH_3$, R_2=Ph, 2,9-dimethyl-4,7-diphenyl-phenanthrolin; dim

Scheme 1. Structure of ligands.

synthesized through oxo abstraction of the corresponding *cis*-dioxomolybdenum(VI) complexes [9]. The complexes were of the form $[MoO(LH)]_n$ and [MoO(LH)(N–N)] where N–N = bipy(2,2′-bipyridine), phen(1,10-phenanthroline) and diacetyldihydrazones. The structure reveals that the molybdenum acceptor centre is present in a distorted octahedral N_3O_2S donor environment. Schiff-base ligands with ONO-donors are of particular interest because chelation may occur either from ON or ONO sites. Under reduced pressure, the two complexes $CrO_2(CO)_2(shaH_2)$ and $W(CO)_2(shaH)_2$ were isolated from the reaction of $M(CO)_6$ (M = Cr and W) with the tridentate Schiff-base, *N*-salicylidene-2-hydroxyaniline ($shaH_2$) [5]. $ShaH_2$ was bidentate through an oxygen atom of a hydroxyl group and the nitrogen atom of the imine group. Reaction of $Mo(CO)_6$ with $shaH_2$ in air gave the oxomolybdenum complexes MoO(sha) and $Mo_2O_4(sha)_2$, depending on the time of reaction [10]. Recently, we reported that the reaction of the tridentate Schiff-base salicylideneimine-2-anisole (salanH) with $Mo(CO)_6$ resulted in formation of the dinuclear oxo complex $Mo_2O_4(salan)_2$ [11]. In this article, we describe the reactions of molybdenum hexacarbonyl with salanH in presence of one of the heterocyclic nitrogen ligands 2,2′-bipyridine, 1,10- phenanthroline and 2,9-dimethyl-4,7-diphenyl-phenanthroline. Scheme 1 represents the structures of the Schiff-base (salanH) and the heterocyclic nitrogen ligands used in the reactions.

2. Experimental

$Mo(CO)_6$, 2,2′-bipyridine, 1,10-phenthroline and 2,9-dimethyl-4,7-diphenylphenanthroline were purchased from Aldrich. Salicylideneimine-2-anisole was prepared as described in the literature [12]. All solvents used are of analytical grade and purified by distillation according to standard methods.

Table 1. Elemental analysis and mass spectrometry data for molybdenum complexes.

Complex	%C Found (Calcd)	%H Found (Calcd)	%N Found (Calcd)	Mass spectrometry Molecular weight	*m*/*z*
$[O_2(\mathrm{salanH})\mathrm{Mo}(\mu\text{-}O)_2\mathrm{MoO_2bpy}]$	42.7(42.9)	3.0(3.1)	6.3(6.2)	671.45	672 $[P^+]$
$[O_2(\mathrm{salanH})\mathrm{Mo}(\mu\text{-}O)_2\mathrm{MoO_2phen}]$	44.8(44.9)	3.1(3.0)	6.1(6.0)	695.48	696 $[P^+]$
$[O_2(\mathrm{salanH})\mathrm{Mo}(\mu\text{-}O)_2\mathrm{MoO_2dim}]$	55.1(54.9)	3.9(3.8)	4.6(4.8)	875.73	876 $[P^+]$
$[\mathrm{Mo(CO)_4(salanH)}]\cdot\mathrm{bpy}$	56.7(56.9)	3.4(3.6)	6.9(7.1)	591.50	564 $[P\text{–}CO]^+$
$[\mathrm{Mo(CO)_4(salanH)}]\cdot\mathrm{phen}$	58.3(58.5)	3.5(3.4)	6.7(6.8)	615.52	588 $[P\text{–}CO]^+$
$[\mathrm{Mo(CO)_4(salanH)}]\cdot\mathrm{dim}$	66.2(66.4)	4.0(4.2)	5.5(5.3)	795.77	768 $[P\text{–}CO]^+$

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. The spectra of the complexes in diferent solvents (DMSO, benzene, THF, ethanol and CH_2Cl_2) were of concentrations ca 1×10^{-5} M. ^{1}H NMR measurements were performed on a Varian-Mercury 300 MHz spectrometer. Samples were dissolved in $(CD_3)_2SO$ with TMS as internal reference. Thermogravimetric (TG) analysis was carried out under N_2 with a heating rate of $10^\circ\mathrm{C\,min^{-1}}$ using a Shimadzu DT-50 thermal analyzer. The complexes were also characterized by elemental analysis (Perkin-Elmer 2400 CHN elemental analyzer) and mass spectrometry. Table 1 gives the elemental analysis and mass spectrometry data for the complexes.

2.1. *Syntheses of [O_2(salanH)Mo(μ-O)$_2$MoO$_2$L]*

A mixture of $Mo(CO)_6$ (0.1 g, 0.37 mmol), salanH (0.08 g, 0.37 mmol) and L, bpy (0.06 g, 0.37 mmol); or phen (0.07 g, 0.37 mmol); or dim (0.14 g, 0.37 mmol) in THF (ca 30 cm^3) was heated to reflux in air for 6–10 h. The solution changed from yellow, orange, red and finally to reddish-brown. The reaction mixture was cooled and the solvent was removed on a vacuum line. The complex was washed several times with boiling petroleum ether and then recrystallized from DMF. The bpy derivative (75% yield) is dark brown while that of the phen and dim derivatives are light brown with 90 and 50% yield, respectively.

2.2. *Syntheses of [Mo(CO)$_4$(salanH)](L) complexes*

$Mo(CO)_6$ (0.1 g, 0.37 mmol), salanH (0.08 g, 0.37 mmol) and L, bpy (0.06 g, 0.37 mmol); or phen (0.07 g, 0.37 mmol); or dim (0.14 g, 0.37 mmol) were mixed in a sealed tube containing THF (ca 30 cm^3). The mixture was degassed with one freeze-thaw cycle and then heated for 24 h. $[\mathrm{Mo(CO)_4(salanH)}]\cdot$phen gave a light brown precipitate with a yield of 80% while the other two complexes $[\mathrm{Mo(CO)_4(salanH)}]\cdot$bpy and $[\mathrm{Mo(CO)_4(salanH)}]\cdot$dim gave dark red precipitates in 53 and 75% yield, respectively.

3. Results and discussion

3.1. *IR and ^{1}H NMR spectra*

Reactions of $Mo(CO)_6$ with salicylideneimine-2-anisole (salanH) in the presence of a secondary ligand L (L = 2,2′-bipyridine, bpy; 1,10-phenanthroline, phen or 2,9-dimethy1-4,7-diphenylphenanthrolin, dim) in air resulted in the formation of dinuclear complexes $[O_2(salanH)Mo(\mu\text{-}O)_2MoO_2L]$. Under reduced pressure, the corresponding reactions gave tetracarbonyl derivatives $[Mo(CO)_4(salanH)](L)$. The IR spectrum of salanH displayed bands due to the functional groups ν(OH), ν(C=N), ν(C–OH) and ν(C–OCH$_3$) at 3433, 1612, 1242 and 1149 cm^{-1}, respectively. The IR spectra of the complexes showed the ligand bands with the appropriate shifts due to complex formation (table 2). The IR spectra of the dinuclear complexes displayed the

Table 2. Important IR data for salanH, bpy, phen, dim and its molybdenum complexes.

	IR data (cm^{-1})[a]							
Compound	ν(OH)	ν(CO)	ν(C=N)	ν(CO)	ν(C–OCH$_3$)	δ(py)	ν(Mo–O)	ν(Mo–N)
SalanH	3433	–	1612	1242	1149	–	–	
bpy	–	–	1574 1550s 1524sh	–	–	644m 617m 518w	–	
$[O_2(salanH)Mo(\mu\text{-}O)_2MoO_2bpy]$[b]	3420b	–	1601s 1574m 1550m	1230m	1100m	730m 671s 598m	530m	421m
$[Mo(CO)_4(salanH)] \cdot bpy$[c]	3422b	2010s 1912s 1871s 1813s	1597m 1562m 1524m	1226m	1112m	730s 650m 598m	552m	421m
dim	–	–	1620s 1574s 1540s	–	–	625m 579m 540m	–	–
$[O_2(salanH)Mo(\mu\text{-}O)_2MoO_2dim]$[d]	3417b	–	1620s 1575s 1547m	1220m	1112m	680m 640m 569m	550w	448w
$[Mo(CO)_4(salanH)] \cdot dim$[e]	3422b	2006s 1882b 1863sh 1828s	1620s 1570s 1543s	1230m	1115m	706s 625m 573w	555mw	486m
phen	–	–	1645m 1622m 1589s 1555m	–	–	694m 617m 590m 500w	–	–
$[O_2(salanH)Mo(\mu\text{-}O)_2MoO_2dim]$[f]	3425b	–	1628m 1617m 1586m	1218m	1100m	718m 625s 613sh 588sh	550m	440m
$[Mo(CO)_4(salanH)] \cdot phen$[g]	3422b	2006s 1900sh 1867s 1825s	1654w 1636w 1627s 1606w	1233m	1112m	725m 690m 648m 609m	549w	425w

[a] s, strong; m, medium; w, weak; b, broad; sh, shoulder.
[b] ν(Mo–O–Mo) = 733, 671 cm^{-1}; ν_s(Mo=O) = 940; ν_{as}(Mo=O) = 903 cm^{-1}.
[c] ν(N $\cdots$ H) = 3020 cm^{-1}.
[d] ν(Mo–O–Mo) = 706, 640 cm^{-1}; ν_{as}(Mo=O) = 961; ν_s(Mo=O) = 888 cm^{-1}.
[e] ν(N $\cdots$ H) = 3024 cm^{-1}.
[f] ν(Mo–O–Mo) = 724, 625 cm^{-1}; ν_s(Mo=O) = 918; ν_{as}(Mo=O) = 887 cm^{-1}.
[g] ν(N $\cdots$ H) = 3019 cm^{-1}.

non-ligand bands at 530–550 and 421–448 cm^{-1} due to Mo–O and Mo–N bonds, respectively (table 2). These spectroscopic data suggested coordination of molybdenum to salanH_2 through the imine–nitrogen atom and the oxygen atom of the methoxy group. Furthermore, the in-plane ring deformation bands of py of the heterocyclic ligands shifted to higher frequencies suggesting bonding through the pyridyl nitrogen [13]. The IR spectra displayed additional stretching frequencies at 918–961 and 887–903 cm^{-1} due to symmetric and asymmetric stretching frequencies of terminal Mo=O bonds for a *cis* MO_2 fragment of a dimeric structure having the core Mo_2O_6 (table 2) [14, 15]. In addition, two stretching frequencies were observed at 706–733 and 625–671 cm^{-1} due to symmetric and asymmetric stretching frequencies of the Mo–O–Mo bonds (table 2) [16]. The IR spectra of the dinuclear complexes showed lower stretching frequencies for the OH bands than the free ligands (table 2) indicating the presence of intramolecular hydrogen bonding which was confirmed by ^{1}H NMR, where higher field shifts for the OH signal at 10.63–10.65 ppm were observed (table 3) [17]. The resonances of the aromatic protons and the protons of pyridyl rings of salanH are listed in table 3. According to the elemental analyses and spectroscopic data, dinuclear complexes might consist of two molybdenum atoms bridged by two oxygen atoms. Each molybdenum is bonded to two *cis* terminal oxygen atoms. One molybdenum atom is coordinated to a salanH ligand through the nitrogen of the imine group and the oxygen of the methoxy group while the other molybdenum is bonded to a heterocyclic ligand, L, through the two nitrogen atoms of the pyridyl rings. The proposed structures of the complexes (**I**, **II**) are shown in scheme 2. Such structures suggest that the molybdenum has a +6 formal oxidation state with a d^0 electronic configuration [18]. The salanH ligand coordinates to molybdenum as a bidentate ligand with free OH group, which formed intramolecular hydrogen bonds with the oxygen atom of M=O moiety.

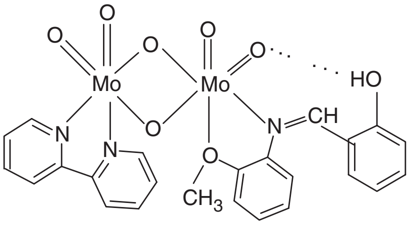
In comparison, reaction of $Mo(CO)_6$ with the Schiff-bases, salicylideneimine-2-anisole (salanH) and *N*-salicylidene-2-hydroxyaniline (shaH_2), resulted in formation of two oxo-molybdenum complexes $Mo_2O_4(salan)_2$ [11] and $Mo_2O_4(sha)_2$ [10], respectively, with the ligands coordinated oxidatively to molybdenum as a tridentate ligand. In the present dinuclear complexes, Mo_2O_6(salanH)L, salanH coordinated to a molybdenum atom as a bidentate ligand leaving the OH group free to hydrogen bond; the second salanH was replaced by the heterocyclic nitrogen ligand.

Under reduced pressure, reaction of salanH with $Mo(CO)_6$ in presence of bpy, phen or dim gave tetracarbonyl complexes with the molecular formula $[Mo(CO)_4(salanH)](L)$. The IR spectra of the complexes displayed characteristic

Table 3. Important ^{1}H NMR data for salanH and its molybdenum complexes.

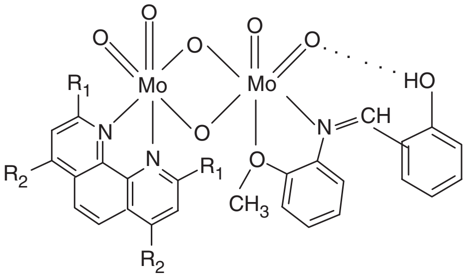
Compound	^{1}H NMR data (ppm)[a]
salanH	7.3(m, Ph), 9.01(s, N=CH), 13.86(s, OH)
$[O_2(salanH)Mo(\mu\text{-}O)_2MoO_2bpy]$	7.4–8.7(m, Ph), 9.23(s, N=CH), 10.63(s, OH)
$[O_2(salanH)Mo(\mu\text{-}O)_2MoO_2phen]$	6.8–8.2(m, Ph), 8.2, 8.5, 9.5 (d,Ph), 9.1(s, N=CH), 10.65(s, OH)
$[O_2(salanH)Mo(\mu\text{-}O)_2MoO_2dim]$	6.8–7.8(m, Ph), 8.90(s, N=CH), 10.65(s, OH)
$[Mo(CO)_4(salanH)] \cdot bpy$	7.5–9.0(m, Ph), 9.24(s, N=CH), 10.22(s, NH), 10.62(s, OH)
$[Mo(CO)_4(salanH)] \cdot phen$	8.1–8.3(m, Ph), 8.9, 9.51 (d,Ph), 9.2(s, N=CH), 10.25(s, NH), 10.68(s, OH)
$[Mo(CO)_4(salanH)] \cdot dim$	7.0–8.8(m, Ph), 9.10(s, N=CH),10.23(s, NH), 10.63(s, OH)

[a] s, singlet; d, doublet; m, multiplet.



I

$[O_2(\text{salanH})Mo(\mu\text{-}O)_2MoO_2(\text{bpy})]$ complex

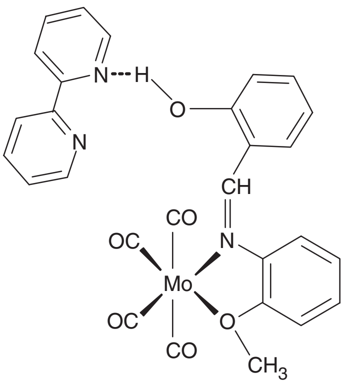


II

$[O_2(\text{salanH})Mo(\mu\text{-}O)_2MoO_2L]$ complex, L = phen or dim

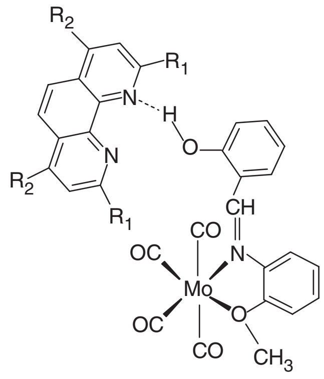
Scheme 2. Proposed structures for complexes I and II.

bands due to C=N, C–O and C–OCH_3 groups of the ligands with the corresponding shifts (table 2). In addition, the IR spectra of the complexes displayed stretching bands due to OH group at lower frequencies than that of the free salanH indicating hydrogen bonding (table 2). Hydrogen bonding was confirmed by ^1H NMR spectra where the OH signal was at a higher field than that of the ligand (table 3). IR spectra of $[Mo(CO)_4(\text{salanH})](L)$ exhibited shifts to lower frequency to give bands at around 3019–3024 cm^{-1} (table 2). This shift could be assigned to the stretching frequency of the $OH \cdots N$ bond [19]. Further evidence was the presence of an NH signal in the ^1H NMR spectra (table 3). Also, the in-plane ring deformation bands of the py moieties of L were shifted to higher frequencies due to bonding of the pyridyl nitrogen. Therefore, the bonding of bpy, phen or dim to the complex is probably through hydrogen bonding between the hydroxyl group of salanH and the nitrogen of the pyridyl moiety. In addition, the IR spectra of the complexes exhibited a pattern of four CO stretching frequencies due to four terminal carbonyl groups attached to the molybdenum atom with $2a_1 + b_1 + b_2$ symmetry (table 2). The previously reported tetracarbonyl complex $Mo(CO)_4(o\text{-}Ph_2PC_6H_4\text{-}CH{=}NEt)$ showed four $\nu(CO)$ bands at 2008, 1892, 1872 and 1856 cm^{-1} [20]. Also, the reactions of $M(CO)_6$, M = Cr, Mo and W, with 2-(2′-pyridyl)benzimidazole (pbiH) in the presence of 2,2′-bipyridine (bpy) gave the tetracarbonyl derivatives $[M(CO)_4(\text{pbiH})] \cdot \text{bpy}$ whose IR spectrum displayed four stretching frequencies due to the carbonyl groups (2014, 1911, 1867, and 1813 cm^{-1}).



III

$[Mo(CO)_4(salanH)]\cdot bpy$



IV

$[Mo(CO)_4(salanH)]\cdot phen$ $R_1 = R_2 = H$

$[Mo(CO)_4(salanH)]\cdot dim$ complex $R_1 = CH_3$, $R_2 = Ph$

Scheme 3. Proposed structures for complexes III and IV.

Elemental analysis and spectroscopic data suggested the structures shown in scheme 3 for the molybdenum tetracarbonyl complexes (**III**, **IV**).

3.2. *Electronic absorption spectra*

Electronic absorption spectra of salanH and its molybdenum complexes were investigated in DMSO, benzene, THF, EtOH and CH_2Cl_2. Two absorption bands were observed for salanH at 280–294 and 340–348 nm due to π–π^* and η–π^* transitions, respectively. The dinuclear molybdenum complexes are insoluble in benzene, THF, EtOH and CH_2Cl_2 but are soluble in polar aprotic coordinating

Table 4. UV-Vis data for salanH and molybdenum complexes in different solvents.

Compound	λ (nm)[a] DMSO	Benzene	THF	EtOH	CH_2Cl_2
SalanH	294 345	285 340	288 349	275 340	280 348
[O_2(salanH)Mo(μ-O)$_2$MoO_2bpy]	288 320(sh)	Insol.	Insol.	Insol.	Insol.
[O_2(salanH)Mo(μ-O)$_2$MoO_2phen]	300 328(sh) 368(sh)	Insol.	Insol.	Insol.	Insol.
[O_2(salanH)Mo(μ-O)$_2$MoO_2dim]	288 358(sh)	Insol.	Insol.	Insol.	Insol.
[Mo(CO)$_4$(salanH)] · bpy	290 420(b)	300 356(b) 494(b)	290 462(b)	294 470(b)	298 384(b) 472(b)
[Mo(CO)$_4$(salanH)] · phen	328(sh) 344(sh)	Insol.	Insol.	Insol.	Insol.
[Mo(CO)$_4$(salanH)] · dim	290 400(b)	288 450(b)	288 440(b)	Insol.	288 412(b)

[a] sh, shoulder; b, broad.

solvents like DMSO. The UV-Vis spectra of dinuclear complexes in DMSO showed a strong band at 288–300 nm (π–π*) with a shoulder at 320–368 nm (n–π*). Both bands exhibited bathochromic shift for the complex [O_2(salanH)Mo(μ-O)$_2$MoO_2phen] and hypsochromic shifts for the other two dinuclear complexes (table 4). The tetracarbonyl complexes showed a band in the UV and a weak broad band in the visible region. The π–π* electronic transitions of these complexes showed bathochromic shifts. In addition, the broad band in the range 400–494 nm, could be due to ligand to metal charge transfer. Previous electronic absorption spectra of M(CO)$_4$L complexes (M = Re, Cr, Mo or W and L = 2,2-bipyridine or a derivative diimine ligand) exhibited a strong absorption band in the UV region and an intense absorption band in the visible region which is strongly solvent dependent and attributed to the MLCT transitions [21–23].

3.3. *Thermogravimetric analysis*

Thermal studies of the dinuclear complexes [O_2(salanH)Mo(μ-O)$_2$MoO_2bpy] (**I**) and [O_2(salanH)Mo(μ-O)$_2$MoO_2phen] (**II**) were carried out using thermogravimetric techniques; their TG and DTG plots display well-defined decomposition steps.

$$\textbf{I}\quad Mo_2(O)_6(C_{14}H_{13}O_2N)(C_{10}H_8N_2) \xrightarrow[-C_7H_4N]{390-610\ K} Mo_2C_{17}H_{17}O_8N_2 \xrightarrow[-C_9H_{12}N_2O_2]{610-730\ K} Mo_2O_6C_8H_5$$

348−528 K

$$\textbf{II}\quad Mo_2(O)_6(C_{14}H_{13}O_2N)(C_{12}H_8N_2) \xrightarrow[-C_3H_4O]{} Mo_2C_{24}H_{21}O_6N_3 \xrightarrow[-C_7H_8N_2]{528-647\ K}$$

$$Mo_2C_{17}H_{13}O_6N \xrightarrow[-C_{12}H_6NO]{647-785\ K} Mo_2O_6C_4H_3$$

The DTG peak of $[O_2(salanH)Mo(\mu\text{-}O)_2MoO_2bpy]$ (**I**) showed two separated peaks corresponding to two successive decomposition steps in the ranges 390–610 and 610–730 K, respectively. The percentage weight losses (15.19 and 26.82%) were attributed to elimination of C_7H_4N and $C_9H_{12}N_2O_2$ to leave the metallic residue $Mo_2O_6C_8H_5$ [24]. The thermal curve of **II** showed two successive decomposition steps in addition to a composite peak with two overlapping peaks. The two successive steps were due to loss of C_3H_4O (8.05%) and $C_7H_8N_2$ (17.27%). The composite peak was in the range 647–785 K. The percentage weight loss corresponded to elimination of $C_{12}H_6NO$ to yield the metallic residue $Mo_2O_6C_4H_3$.

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