



Novel isatinoxime molybdenum and chromium complexes: Synthesis, spectroscopic, and thermal characterization

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HIGHLIGHTS

- Reactions of molybdenum or chromium hexacarbonyls with isatinoxime were studied.
- The prepared isatinoxime ligand and its complexes were characterized.
- Analytical, spectral, molar conductivity, magnetic and thermal measurements were used.
- The prepared complexes are non-electrolytes.
- The binuclear oxo complexes with formulas $[\text{Mo}(\text{H}_2\text{L})(\text{THF})(\text{O})_2]_2$, and $[\text{Cr}(\text{H}_2\text{L})(\text{O})_2]_2$.

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ABSTRACT

Reactions of molybdenum and chromium hexacarbonyls with isatin-3-oxime (H_2L) in THF were carried out under sun light or according to the traditional thermal reflux synthetic routes. Di- μ -oxo bimolybdenum complex, $[\text{Mo}(\text{H}_2\text{L})(\text{THF})(\text{O})_2]_2$ was successfully synthesized under the reflux conditions. However, the dichromium oxo complex, $[\text{Cr}(\text{H}_2\text{L})(\text{O})_2]_2$ was prepared using the sun light route. The prepared complexes were characterized using elemental analysis, IR, mass spectrometry, UV–Vis spectra, thermal analysis, and magnetic measurements.

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1. Introduction

Isatins and their derivatives are well known to have coordinative properties [1–4] and biological activities like antibacterial, antimicrobial, antifungal, and anti-HIV activities [5–9]. It is an endogenous compound that is widely distributed in mammalian tissues and body fluids. Isatin also readily crosses the blood–brain barrier, suggesting its possible action on the central nervous system (CNS) [10]. Consequently, the study of metal complexes of Schiff-base ligands derived from isatin has received much attention during the last years [11–13]. Recently, Isatin-thiosemicarbazone $\text{Cu}(\text{II})$ complexes related to the antiviral drug, methisazone, were studied [14]. Different metal complexes such as nickel, copper, and zinc complexes of isatin and N-methylisatin-3-picolinoyl hydrazone

were synthesized and characterized [15]. However, isatin oxime, as a special type of isatin Schiff-base ligands, has aroused a keen interest because of its simple synthesis by condensation of isatin with hydroxyl amine [16]. Isatin-3-oxime (H_2OXI) has long been known to form complexes, which can undergo significant changes in biological activities on complexation as compared to their pure ligand forms [17–19].

In earlier work we reported the reactions of some metal carbonyls and oxime ligands [20–23], which prompted us to further investigate the reactions of some metal carbonyls such as $\text{Mo}(\text{CO})_6$ and $\text{Cr}(\text{CO})_6$ with isatin-3-oxime. The aim of the present work was to synthesize, and characterize the ligand and its novel complexes with molybdenum, and chromium.

2. Experimental

2.1. Materials and instrumentation

The following were purchased from commercial sources and used without further purification: $\text{Mo}(\text{CO})_6$ (Aldrich), $\text{Cr}(\text{CO})_6$

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(Aldrich), and isatin (Merck). Solvents were of analytical grade and were purified, and dried according to standard procedures [24]. Elemental analyses were carried out on a Perkin-Elmer 2400 CHN elemental analyzer. The electronic spectra were recorded with a Jasco UV-Vis spectrophotometer (Model: V530). The IR spectra were recorded as KBr disks with a Unicam-Mattson 1000 FT-IR spectrometer. Molar conductance measurements were taken by using Jenway 4010 conductivity meter. Magnetic moments were determined on a Sherwood Scientific magnetic moment balance (Model: MK1) at room temperature (25 °C) using with Hg[Co (SCN)₄] as a calibrant. Diamagnetic corrections were applied using Pascal's constants [25]. Mass spectra were performed on a JEOL JMS-AX500 mass spectrometer. Thermogravimetric analyses (TGA) were carried out using a Shimadzu DT-50 thermal analyzer under nitrogen atmosphere with heating rate of 10 °C/min.

2.2. Synthesis of isatin-3-oxime (H₂L) and its metal complexes

2.2.1. Synthesis of isatin-3-oxime (H₂L)

A solution containing 8.5 g (57 mmol) of isatin in 20 mL of ethanol was added dropwise to a stirring solution of 4.0 g (28 mmol) of hydroxylamine in 20 mL of ethanol. The reaction blend was refluxed for 45 min. After cooling, the yellow precipitate oxime was collected by filtration and re-crystallized from ethanol to give a yellow crystalline product (m.p. 239–240 °C, 3.30 g yield, 75%).

Anal. Calc. for C₈H₆N₂O₂ (MW = 162.15): C, 59.62; H, 3.72; N, 17.16. Found: C, 59.20; H, 3.60; N, 17.16.

2.2.2. Synthesis of [Mo(H₂L)(THF)(O₂)₂] complex

Bipyridyl (0.059 g, 0.38 mmol) was added to a solution of Mo(CO)₆ (0.10 g, 0.38 mmol) in 20 mL of THF, with stirring and reflux for 30 min. Isatin-3-oxime (0.0614 g, 0.38 mmol) was dissolved in 5 mL of THF and added to the stirring molybdenum hexacarbonyl solution, then heated to reflux for 9 h. During this time, the color of the reaction blend turned into deep green. The reaction mixture was cooled to room temperature, and then the THF solvent was evaporated by rotatory evaporator. The obtained residue was washed several times with hot petroleum ether (40–60) and with ethyl alcohol to give green solid. The green residue was dried under vacuum for few hours to give a yield of 0.116 g (84%).

Anal. Calc. for C₂₄H₂₈N₄O₁₀Mo₂ (MW = 727.99): C, 39.79; H, 3.90; N, 7.73. Found: C, 39.90; H, 3.84; N, 7.66. Effective magnetic moment, μ_{eff} (BM): 3.31. Molar conductivity ($\Omega^{-1} \text{ mol}^{-1} \text{ cm}^2$): 7.9.

2.2.3. Synthesis of [Cr(H₂L)(O₂)₂] complex

A mixture of Cr(CO)₆ (0.2 g, 0.91 mmol) and isatin-3-oxime (0.147 g, 0.895 mmol) was dissolved in 20 mL of THF. The reaction mixture was left to expose to sunlight for 3 days. The color of the reaction mixture turned into green. The THF solvent was evaporated to give an oily residue, which was washed with hot petroleum ether and then scratched with diethyl ether to finally produce a brown residue. The produced residue was washed several times with ethanol and left to dry under vacuum to give a yield of 0.174 g (78%).

Anal. Calc. for C₁₆H₁₂N₄O₄Cr₂ (MW = 419.95): C, 39.04; H, 2.46; N, 11.38. Found: C, 38.49; H, 2.60; N, 11.30. Effective magnetic moment, μ_{eff} (BM): 1.95. Molar conductivity ($\Omega^{-1} \text{ mol}^{-1} \text{ cm}^2$): 8.5.

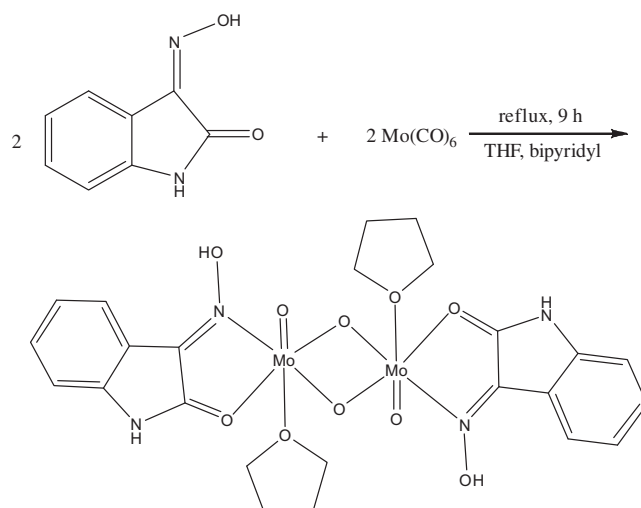
3. Results and discussion

The reaction of molybdenum hexacarbonyl with H₂L ligand in presence of bipyridyl produced the dinuclear complex Mo₂O₄(H₂L)₂(THF)₂ in high yield (Scheme 1). Actually, molybdenum hexacarbonyl did not react with the H₂L ligand in absence of bipyridyl

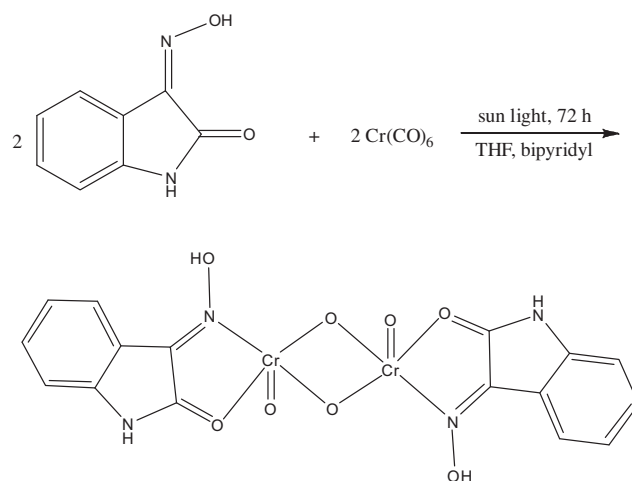
under the atmospheric pressure and gave no product. Consequently, bipyridyl has been used in this reaction because of its labilizing effect on the carbonyl groups [26]. In addition, reactions of metal carbonyls with ligands under atmospheric pressure are complicated by the sublimation metal carbonyls which either result in a small yield or the reaction does not proceed. This problem is avoided by starting with the amine complexes of metal carbonyls [27]. It is worthy to report that, repeating this reaction in sunlight did not proceed.

On the other hand, the reaction of chromium hexacarbonyl with H₂L ligand in sunlight gave the dinuclear complex Cr₂O₄(H₂L)₂ in high yield (Scheme 2). Formation of oxomolybdenum and oxochromium complexes may be attributed to the reaction with oxygen of the atmosphere as it was expected [22]. It is noteworthy that repeating this reaction under reflux condition did not proceed.

The complexes were initially characterized on the basis of their mass spectra and elemental analyses. The mass spectra of the molybdenum and chromium complexes are shown Fig. 1. For the molybdenum complex, it revealed peaks (m/z) at 363.9, 162, 161.7, 155, 145.9, and 129.9, which may be attributed to 1/2 (Mo₂O₄(H₂L)₂(THF)₂), H₂L, MoO₄, MoO₂CN, MoO₃, and MoO₂ fragments, respectively. On the other hand, the mass spectrum of the chromium complex exhibits peaks (m/z) at 329.9, 162, and 116,



Scheme 1. Proposed structural formula for the dimolybdenum complex.



Scheme 2. Proposed structural formula for the dichromium complex.

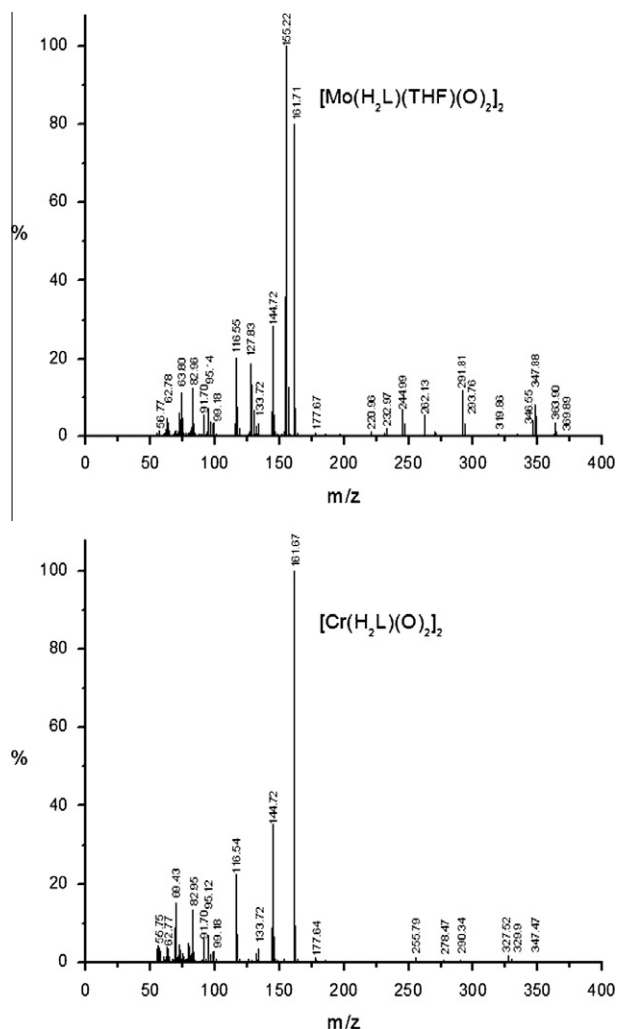


Fig. 1. The mass spectra for $[\text{Mo}(\text{H}_2\text{L})(\text{THF})(\text{O})_2]_2$ and $[\text{Cr}(\text{H}_2\text{L})(\text{O})_2]_2$ complexes.

which may be assigned to $\text{Cr}_2\text{O}_4(\text{H}_2\text{L})_2\text{--H}_2\text{L}$, H_2L , and CrO_4 , respectively.

It is noteworthy to report that the complexes described in this study are soluble in DMF and DMSO. Solutions of the complexes in DMSO with concentration of 1×10^{-3} M have molar conductivity (λ_m) of 7.9 and $8.5 \Omega^{-1} \text{mol}^{-1} \text{cm}^2$ for the molybdenum and chromium complexes, respectively, and these values indicate the non-electrolytic nature of the prepared complexes [28,29].

Analyzing the IR spectra of the synthesized complexes was useful in assigning the ligand coordination modes to the molybdenum and chromium centers. The IR spectra of the H_2L ligand and its molybdenum and chromium complexes as well as shown in Fig. 2. The IR spectrum of the free ligand displayed some characteristic absorption peaks at 3228, 3189, 3090, 1714, 1661, 946 cm^{-1} , which may be due to OH, NH, C–H aromatic, C=O, C=N and N–O stretching vibrations, respectively [30,31]. Generally, the IR absorption bands of O–H, C=N and N–O stretching vibrations are used to assign the coordination modes of the C–N–OH group to the metal center. Upon coordination through the N atom of the oxime group, the stretching vibration corresponding to C=N shifts to lower frequency. In contrast, the bands corresponding to O–H and N–O stretching vibrations shift to higher frequencies [30,32]. The molybdenum and chromium complexes displayed the peaks corresponding to C=N stretching vibrations of the oxime group at 1602 and 1624 cm^{-1} , respectively. These bands have shifted to lower frequencies of 59 and 37 cm^{-1} for the molybdenum and chromium

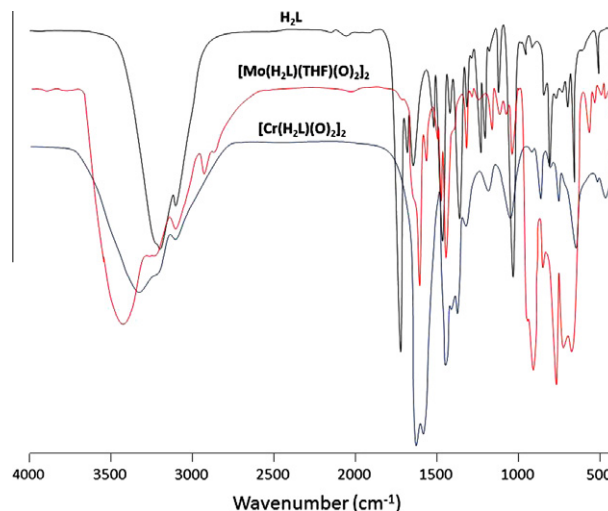


Fig. 2. The IR spectra of H_2L ligand and its molybdenum and chromium complexes.

complexes, respectively. In contrast the peaks corresponding to O–H and N–O stretching vibrations were observed at 3425 and 1038 cm^{-1} for the molybdenum complex and at 3325 and 1024 cm^{-1} for the chromium complex. Such behavior evidently indicates that the isatin oxime ligands are coordinate to molybdenum centers and chromium centers, in the molybdenum and chromium complexes, respectively, through the nitrogen atom of the C=N–OH group [22,30,33]. The IR spectrum the molybdenum and chromium complexes revealed one band for each complex, corresponding to the M=O asymmetric vibration, and observed at 904 and 871 cm^{-1} , respectively. The absence of M=O symmetric stretching vibration may suggest a symmetrical molecule in which the two M=O moieties are located in a *trans* positions to one another. These results are in good agreement with those observed for $\text{Mo}_2\text{O}_2(\text{HBNO})_4$ and $[\text{Mo}_2\text{O}_2(3,5\text{-DBQ})]_2$ [22,34]. The dinuclear structure of the molybdenum and chromium complexes may be inferred from the IR spectra. The IR spectra of $[\text{Mo}(\text{H}_2\text{L})(\text{THF})(\text{O})_2]_2$ and $[\text{Cr}(\text{H}_2\text{L})(\text{O})_2]_2$ revealed absorption bands, that are not present in the spectrum of the free H_2L ligand, at 763 and 750 cm^{-1} , respectively, and may be assigned to asymmetric stretching vibrations associated with $\text{M}_2(\mu\text{-O})_2$ structural unit. Consistence with this assignment, the IR spectrum of the structurally characterized dinuclear, doubly-bridged $[\text{Mo}_2\text{O}_4(\text{Sabb})_2]$ complex showed an absorption peak at 760 cm^{-1} due to the stretching frequency of the bridging M–O [35]. Also, $[\text{Au}_2(\text{bipy})_2(\mu\text{-O})_2]^{2+}$ and $[(\text{Cu}_2(\text{Sp})_2(\mu\text{-O})_2)]^{2+}$ complexes were found to exhibit the $\mu(\text{M}_2\text{O}_2)$ at 682 and 619 cm^{-1} , respectively [36]. Additional characteristic vibrations of coordinated THF are observed for the molybdenum complex at 2922 and 2866 cm^{-1} (C–H stretching vibrations) and 1116 cm^{-1} (vibration associated with C–O–C moiety). This assignment is in good agreement with that reported for coordinated THF [30,37,38]. Finally, stretching vibrations which can be assigned to M–O and M–N were found respectively, at 659 and 513 cm^{-1} for the molybdenum complex and at 630 and 463 cm^{-1} for the chromium complex [36].

The magnetic susceptibility measurements of the synthesized complexes were carried out on the solid samples at 298 K. The effective magnetic moment (μ_{eff}) was found to be 3.31 and 1.95 B.M. for the dimolybdenum and dichromium complexes, respectively. For a dimolybdenum(IV) complex, an intramolecular magnetic exchange can produce $S = 0$, $S = 1$ and $S = 2$ magnetic state[22]. When the magnetic exchange was antiferromagnetic, the $S = 0$ would be the ground state. It was reported that the dimolybdenum(IV) complexes containing Mo–Mo bond are diamagnetic

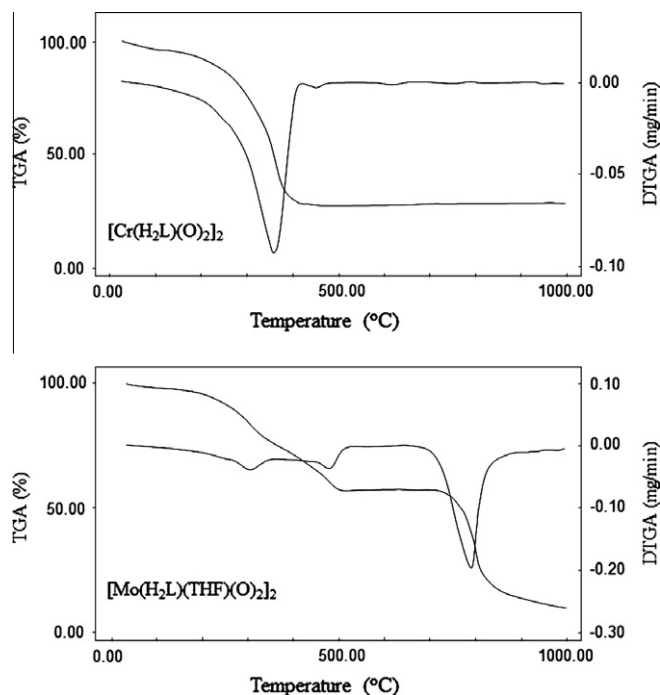


Fig. 3. TGA and DTGA plots for Chromium and Molybdenum complexes.

due to molybdenum–molybdenum bonding interaction. Moreover, oxo-bridged metal ion dimers also exhibit magnetic exchange interactions [22,39]. Consequently, the obtained magnetic for $\text{Mo}_2\text{O}_4(\text{H}_2\text{L})_2(\text{THF})_2$ and $\text{Cr}_2\text{O}_4(\text{H}_2\text{L})_2$ complexes can fit with the proposed dimeric structures containing two M(IV) centers and bonded through two M–O–M bridges. Additionally, as expected, the decrease in the effective magnetic moments for both dimetallic complexes than the spin-only value (4.9 BM) was theoretically calculated for four unpaired electrons and may be due to the intermolecular magnetic interaction between the two M(IV) centers through the two M–O–M bridges. Also, the magnetic moment value for the dichromium complex was lower than that of the dimolybdenum complex, which mean that the unpaired electrons on the two chromium centers can interact more than those of the two molybdenum centers due to the fact that the atomic orbitals of molybdenum atom are larger than those of chromium atom so that the probability that the electrons can see each other is more for the dichromium atoms than that for the dimolybdenum atoms [40].

Table 1

The electronic absorption data for H_2L ligand and its complexes in recorded DMSO.

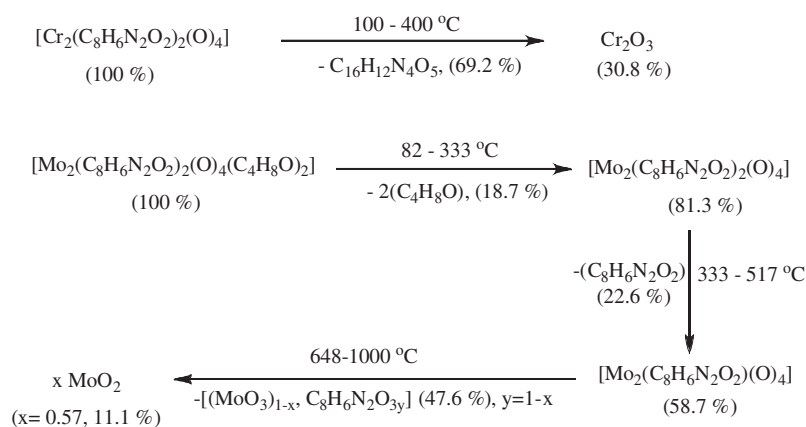
| Compound | λ (nm; $\log \epsilon^a$) |
|---|-------------------------------------|
| H_2L | 300 (3.88), 400 ^b (2.10) |
| $[\text{Cr}(\text{H}_2\text{L})(\text{O})_2]_2$ | 350 (4.16), 600 ^b (2.80) |
| $[\text{Mo}(\text{H}_2\text{L})(\text{THF})(\text{O})_2]_2$ | 310 (4.22), 350 ^b (3.67) |

^a $\epsilon \text{ M}^{-1} \text{ cm}^{-1}$.

^b Broad.

The thermal studies of the dimolybdenum and dichromium complexes shown in Fig. 3 provided further insight into the proposed structures and the thermal stability of the complexes investigated. The reported complexes were found to be air stable. The thermogravimetric studies were carried out within the temperature range 20–1000 °C at a heating rate of 10 °C/min under nitrogen atmosphere. The TGA curve of the dichromium complex revealed one decomposition step (Scheme 3). This step occurred at 101–472 °C and attributed to the decomposition of the organic part of the dichromium complex leaving Cr_2O_3 residue (found 30.88%, calcd. 30.8%). On the other hand, the thermal decomposition of dimolybdenum complex took place in three steps as showed by the TGA analysis (Scheme 3). The first step, within the temperature range of 82–333 °C, was related to the evolution of two THF molecules due to a mass loss of 19.7% (calcd. 18.7%). The second step occurred in the temperature range of 333–517 °C, and was accompanied by a weight loss of 22.5% (calcd. 22.3%), corresponding to the loss of one isatin-oxime ligand molecule. The third stage involved a significant mass loss (47.6%) extending from 648 °C to 1000 °C, corresponding to the removal of one molecule ligand to give a molybdenum dioxide residue ($x \text{ MoO}_2$; 10%, where $x = 0.57$) and sublimed $(1 - x)\text{MoO}_3$ molecule.

The electronic absorption spectra recorded for the two complexes and the free ligand in DMSO are summarized in Table 1. The free ligand revealed two absorptions at 300 and 400 nm attributing to $\pi-\pi^*$ and $n-\pi^*$ transitions, respectively. However, the UV–Vis region is characterized by two absorptions displayed by the two prepared complexes. For the dichromium complex, the intense absorption at shorter wave length (350 nm) can be assigned to a $\pi-\pi^*$ transition, while the absorption at the longer wave length (600 nm) may be attributed to a chromium(IV) $^3\text{T}_{1g}-^3\text{T}_{2g}$ transition [41], and/or $n-\pi^*$ transition, which commonly displays a bathochromic shift upon complexation. The higher energy d–d transitions characteristic for Cr(IV) centers are obscured by the intense $\pi-\pi^*$ transition. On the other hand, the dimolybdenum complex showed the absorption corresponding to the intraligand $\pi-\pi^*$ transition at 310 nm. In addition, an intense absorption was



Scheme 3. Proposed thermal decomposition mechanisms of chromium and molybdenum complexes.

observed at 350 nm ($\log \varepsilon = 3.67$) which could be assigned to the oxygen-to molybdenum charge transfer transition. This assignment is in good agreement with that observed for the analogous α -benzoin oxime complex, $\text{Mo}_2\text{O}_4(\text{HBNO})_2$ [22]. Also, the electronic absorption spectrum of the doubly-bridged $[\text{Mo}_2\text{O}_4(\text{Sabb})_2]$ showed the absorption band at 428 nm ($\log \varepsilon = 4.63$) due to the charge transfer transition from oxygen orbital to a metal d-orbital [35].

4. Conclusions

Two novel bimetallic complexes; $[\text{Mo}(\text{H}_2\text{L})(\text{THF})(\text{O})_2]_2$, and $[\text{Cr}(\text{H}_2\text{L})(\text{O})_2]_2$ were prepared using their corresponding metal hexacarbonyls in THF under reflux and sun light routes, respectively. These two complexes were characterized using IR, mass spectroscopy, UV–vis spectra, thermal analysis and magnetism. Additionally, for both complexes, the lowering in μ_{eff} values may be due to strong antiferromagnetic coupling between the two metal centers through the oxo-bridge.

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