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Cis-Dioxomolybdenum (VI) Complexes with N-donor Macrocyclic Ligands

Ramesh Kumar Gautam [1], Chandra Prakash Singh [2], Devendra Pratap Rao [1,*]

[1] Department of Chemistry, D.A-V. (P.G.) College, Kanpur-208001, U.P., India
[2] Department of Chemistry, D.B.S. College, Kanpur – 208006, U.P., India
* Correspondence: devendraprataprao@yahoo.com (D.P.R.);
Scopus Author ID 35119371500

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Abstract: The preparation of one monomeric $[MoO_2(L)](acac)_2$ having a Schiff base came by condensation of di-2-furanylethanedione with 2,3-diaminotoluene and 4 different complexes having formulation $[MoO_2(ML)](acac)_2$, from the result of $[MoO_2(L)](acac)_2$ with 1,3-diketones will be described with this paper. The preparation of each of these five MoO_2 (VI) complexes was symbolized by thermal studies, UV-Vis, IR, elemental analyses, NMR, and molar conductance. The coordination number of molybdenum metal will be six. All 5 complexes of MoO_2(VI) have a geometry of distorted octahedral arrangement. The octahedral arrangement of Mo-metal in prepared complexes is finished by 2 oxo O-atoms and 4 N-atoms from the synthesized ligand. All of the synthesized complexes demonstrated moderate action against S. *aureus* and S. *typhi*. The progression inside the antibacterial task being defined on the thought of chelation speculation.

Keywords: dioxomolybdenum(VI); di-2-furanylethanedione;1,3-diketones;macrocyclic complexes; Schiff base.

1. Introduction

The macrocyclic complex is a cyclic complex that contains at least 9 atoms, including all heteroatoms. In such complexes, at least 3 donor atoms will be present. The discipline of the chemistry of macrocyclic complexes possesses experienced development amid the prior past hundred years [1-5]. Ligands containing N-atom and Schiff bases and their complexes assumed a significant job in improving coordination chemistry. Several research papers have been published, ranging from physicochemical and biochemically pertinent be taught of metal complexes and driven extensive selection of applications [6-11]. Schiff-based compounds were applied as medicines and had worthwhile antifungal, antiviral, anti-inflammatory, and antitumor properties [12-20]. Metal ions catalyzed the transamination reactions via the production of transitional Schiff bases formulated with vitamin B6 [15].

Fascination with complexes of Schiff base in the bioinorganic field has directed at the purpose of such complexes in imparting manufactured models to the metal-containing positions in metalloproteins and enzymes [21]. Various Schiff bases have anticancer activity of that particular metal complex is greater in evaluation to the isolated ligand [22-24]. They endure durable catalytic fascination with various reactions in chemistry so when memory storage gizmos in consumer electronics [25-29].

Transition metal complexes with extreme denticity ligands have different configurations and coordination numbers [31]. Molybdenum can be adaptable due to its multiple oxidation states, ranging from –2 to +6. In the same way, coordination numbers vary from 4 to 8 [32]. The capability to connect molybdenum complexes with ligands containing N, O- and S-atoms induced growth of Mo Schiff base edifices, which are productive impetuses in homogeneous and heterogeneous reactions [33-39]. MoO_2(VI) complexes with multidentate ligands have super value in the theoretical and functional region predominantly for biological functions. Mo(VI) can be acquired as an effortless $[MoO_4]^{2-}$ ion in an aqueous medium. $[MoO_4]^{2-}$ ion can be based on the concentration and pH of the solution. The $[MoO_4]^{2-}$ ion can perform as an oxygen transfer template [40-41]. Their oxygen transfer qualities play a negative role in the performing mechanism of molybdenum oxotransferase [42,43]. Several redox enzymes are present in a fully oxidized state. In such cases, their active sites consist of a *cis*-dioxomolybdenum moiety [44-46].

In the second series of transition metals, entirely molybdenum is considered a biometal crucial for human, animal, and vegetation pathogenic microorganisms [47,48]. The coordination chemistry of Mo(VI) is of thriving interest for the present research because of their catalytic activities and biological properties [49-52]. Physiological functions of oxomolybdoenzymes are set up via molybdenum [53-55]. Di-2-furanylethanedione may be a versatile chelating agent. Di-2-furanylethanedione has two reactive carbonyl groups, which might be capable of undergoing Schiff base condensation with several di- and polyamines. Therefore, di-2-furanylethanedione has performed an essential function within the synthesis of macrocyclic ligands.

With on pinnacle of context, some dioxomolybdenum(VI) complexes with excessive denticity ligands can synthesize from the condensation of di-2-furanylethanedione with a diamine. The synthesized complex has the ability to undergoing cyclization with 1,3-diketones via the metal template impact. They can be prepared, characterized and their provisional structures are supported by molar conductivity, elemental analysis, electronic, infrared, and nuclear magnetic resonance spectroscopy.

Retaining because of the importance of Mo_2(VI) cations, a new sequence of dioxomolybdenum(VI) macrocyclic complexes were synthesized. Those dioxomolybdenum(VI) macrocyclic complexes with new chelate ligands derived from the condensation of di-2-furanylethanedione with 2,3-diaminotoluene successful in presenting process cyclization with 1,3-diketones through the metallic template impact have been synthesized. Herein, the synthesis and ascertaining structures based totally on molar conductivity, elemental analyses, electronic IR, NMR, and TGA/DTA are reported.

2. Materials and Methods

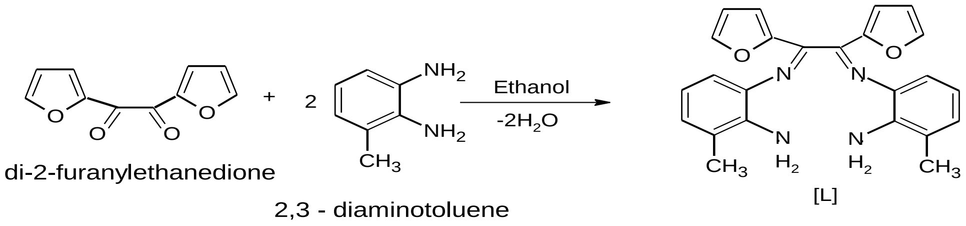
All chemicals utilized for the preparation of Schiff base (ligands) and complexes had been of reagent grade and utilized as obtained from business resources. Molybdenyl acetylacetonate, 2,3-diaminotoluene, di-2-furanylethanedione and 1,3-diketones (2,4-pentanedione, 1-phenyl-1,3-butanedione, 4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedione, and 1,3-diphenyl-1,3-propanedione) were obtained from Aldrich and utilized without similarly purging.

2.1. Analytical methods and physical measurements.

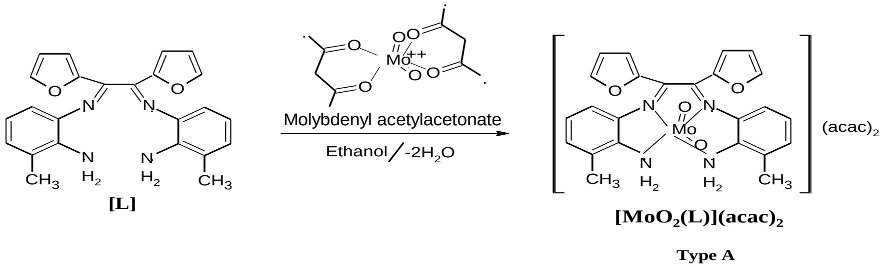
Examinations of C, H and N for the complexes were done at important CRF, NERIST, Nirjuli, Itanagar, Arunachal Pradesh, India by using CHN analyzer. Kjeldahl's method becomes used to assess nitrogen for the synthesized complexes. After the decay of the complex, molybdenum was assessed gravimetrically via standard technique [56]. Estimation of sulfur progressed toward becoming accomplished as barium sulfate [57]. The general method changed into utilized for deciding uncorrected melting factors with the assistance of sulfuric acid bath. The electronic absorption spectral computation (the UV-Visible spectra) of the complexes was recorded on Labinda-UV 3000^+ UV/VIS spectrophotometer inside the reaches 1100 - 220 nm by utilizing ethanol as solvent at UPTTI Kanpur, U.P., India. The infrared spectra of MoO_2(VI) complexes (4000-400 cm^{-1}) had been recorded in KBr on Perkin-Elmer Spectrum model 10.03.06 spectrophotometers at IIT Kanpur. NMR spectra of MoO_2(VI) complexes were procured on JMM ECS-400 (JEOL) spectrometer with 400 MHz for proton (^{1}H NMR). Thermograph of the produced parent complex [MoO_2(L)](acac)$_2$ was done under nitrogen atmosphere in the temperature extend 50 - 600 ^{0}C at the heating rate 10 ^{0}C min^{-1} utilizing TG/DTA - Perkin Elmer, USA thermal analyzers.

2.2. In-situ synthesis of dioxomolybdenum(VI) complexes with ligands derived by condensation of di-2-furanylethanedione with 2,3-diaminotoluene with 1,3-diketones.

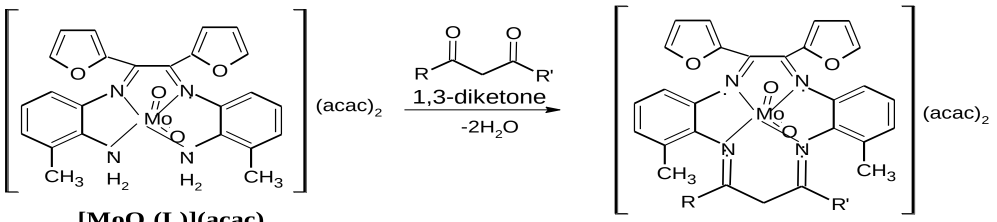
Schemes 1, 2and 3 display the synthesis way of [MoO_2(ML)](acac)$_2$. An ethanolic solution of molybdenyl acetylacetonate (2.5 mmol, 0.81537 g) was brought drop smart to a refluxing solution of di-2-furanylethanedione (2.5 mmol, 0.5570 g) and 2,3-diaminotoluene (5 mmol, 0.61164 g) in ethanol (50 mL) in RB flask. The resulting reaction mixture was slightly refluxed for 3 h. The coloration of the solution becomes brown. The received solid product was filtered off, washed with ethanol, and isolated beneath in vacuo over silica gel. TLC method was employed for checking the purity of the prepared complex. The yield became 46 % (type A).



Scheme 1. Synthesis of Ligand (L).



Scheme 2. Synthesis of [MoO_2(L)](acac)$_2$.



$[MoO_2(L)](acac)_2$

Type A

$[MoO_2(ML)](acac)_2$

Type B

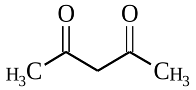
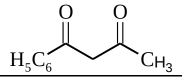
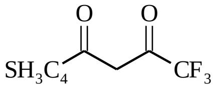
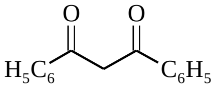
Scheme 3. Synthesis of $[MoO_2(ML)](acac)_2$ where, L= difuranylethanedione+2,3-diaminotoluene; ML= macrocyclic ligands carried out from condensation of L with 1,3-diketones in presence of dioxomolybdenum(VI) cation.

Where, L = di-2-furanylethanedione + 2,3-diaminotoluene; ML = macrocyclic ligands carried out from condensation of L with 1,3-diketones in presence of dioxmolybdenum(VI) cation.

Table 1. Physical and analytical data of the ligand and complexes.

Complex	Empirical Formula	F.W.	Yield (%)	m.p. (^{0}C)	C% Calcd. (found)	H% Calcd. (found)	N% Calcd. (found)	Mo% Calcd. (found)	S% Calcd. (found)
L	$C_{24}H_{22}N_4S_2$	430.59	45	115	66.94 (66.93)	5.14 (5.15)	13.01 (13.00)	--	14.89 (14.87)
$[MoO_2(L)](acac)_2$	$C_{34}H_{36}N_4MoS_2O_6$	756.75	50	125	53.96 (53.98)	4.79 (4.78)	7.40 (7.41)	12.67 (12.64)	8.47 (8.46)
$[MoO_2(ML^1)](acac)_2$	$C_{39}H_{40}N_4MoS_2O_6$	820.84	52	110	57.06 (57.05)	4.91 (4.91)	6.82 (6.81)	11.68 (11.69)	7.81 (7.80)
$[MoO_2(ML^2)](acac)_2$	$C_{44}H_{42}N_4MoS_2O_6$	882.91	53	120	59.85 (59.86)	4.79 (4.78)	6.34 (6.34)	10.86 (10.85)	7.26 (7.24)
$[MoO_2(ML^3)](acac)_2$	$C_{42}H_{37}N_4MoO_6S_3F_3$	942.91	58	118	53.50 (53.48)	3.95 (3.96)	5.94 (5.95)	10.17 (10.16)	10.20 (10.22)
$[MoO_2(ML^4)](acac)_2$	$C_{49}H_{44}N_4MoS_2O_6$	944.98	55	122	62.28 (62.29)	4.69 (4.67)	5.92 (5.90)	10.15 (10.14)	6.78 (6.75)

Table 1'. Description.

For macrocyclic	R	R'	1,3-diketone	Structure
ML^1	CH_3	CH_3	2,4-pentanedione	
ML^2	C_6H_5	CH_3	1-phenyl-1,3-butanedione	
ML^3	C_4H_3S	CF_3	4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedione	
ML^4	C_6H_5	C_6H_5	1,3-diphenyl-1,3-propanedione	

L= ligand derived via condensation of di-2-furanylethanedione with 2,3-diaminotoluene (1:2);
ML^1 = macrocyclic ligand derived via condensation of ligand (L) with 1,3-diketone-2,4-pentanedione ;
ML^2 = macrocyclic ligand derived via condensation of ligand (L) with 1,3-diketone-1-phenyl-1,3-butanedione;
ML^3 = macrocyclic ligand derived via condensation of ligand (L) with 1,3-diketone-4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedione;
ML^4 = macrocyclic ligand derived via condensation of ligand (L) with 1,3-diketone-1,3-diphenyl-1,3-propanedione.

The solution of Kind A suspended in ethyl alcohol uniformly reacted for three h with 1,3-diketones viz. 2,4-pentanedione, 1-phenyl-1,3-butanedione, 4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedione or 1,3-diphenyl-1,3-propanedione (1:1) to gain macrocyclic dirty yellow solid stable products (type B). The purity of the macrocyclic complexes turns into checked by way of TLC.

Elemental analyses (Table 1) of the complexes revealed 1:1 metal to ligand stoichiometry.

2.3. Antibacterial activity assay.

The antibacterial interest of the synthesized dioxomolybdenum(VI) complexes was examined *in vitro* against four bacterial strains, i.e., *Staphylococcus aureus, Bacillus subtilis, Enterobacter aerogene,* and *Salmonella typhi,* utilizing a cup and agar-well diffusion method [58-60]. Doxycycline drug changed into used as the identical general antibacterial agent. Wells having dimensions of 6 mm in diameter were dug inside the agar media with the help of a metallic borer. The density of each bacterial suspension in each well turned into adjusted to 3×10^5 colony-forming units (CFU) mL^{-1}. The standardized suspensions were expanded on the surface of the agar. The produced complexes have been dissolved in 1 % DMSO, and the concentration of the examined sample was 300 μg mL^{-1}. The examined samples were added within the corresponding wells. The rest wells were packed with DMSO and antibacterial medium doxycycline (0.05 %). Growth inhibition becomes tested after 30 h incubation at 35 ^oC.

3. Results and Discussion

3.1. Infrared spectra.

The dioxomolybdeum(VI) had been prepared via the use of an *in-situ* approach to reflux the reaction mixture of di-2-furanylethanedione 2,3-diaminotoluene, and molybdenyl acetylacetonate in 1:2:1 molar ratio in aqueous ethanol, which results in macrocyclic complexes in the scheme. Remarkable infrared spectral bands of the isolated ligand and the MoO_2(VI) complexes and their provisional assignments are given in table 2**.** The bonding of N-atoms of groups azomethine to the molybdenum in all macrocyclic complexes was evidenced with the aid of using the move of $\nu_{C=N}$ to lower frequencies [61-64]. The spectral bands place 1604-1645 cm^{-1}arecoupled to >C=N absorption, which usually seems at 1665 cm^{-1} in isolated ligands [61-63]. The new band at around 508-591 cm^{-1}may be assigned to ν_{Mo-N} vibration [65], i.e., not present in free ligands. The bonding of both keto groups of di-2-furanylethanedione via carbonyl oxygen with diamines becomes supported via the presence of >C=N band and the absence of the > C=O band around 1710 cm^{-1} [66,67]. Infrared spectra of the isolated ligand and its complexes of dioxomolybdeum(VI) are complex because of the appearance of diverse ring vibrations and C-H vibrations. A wideband targeted at 3433 cm^{-1} for $\nu_{asym\ (N-H)}$ and 3065 cm^{-1} for $\nu_{sym\ (N-H)}$. Within the complex $[MoO_2(L)](acac)_2$ bands live unchanged however absent in complex $[MoO_2(ML)](acac)_2$ because this implies non-participation of the NH group in the bonding [68]. The dioxomolybdenum(VI) complexes create a *cis*-dioxo group preferentially because of the d-orbital for bonding. The dioxomolybdenum(VI) complexes revealed two Mo=O stretching bands at 893-910 cm^{-1} and 964-981 cm^{-1} due to asymmetric and symmetric stretching vibrations of the *cis*-$[MoO_2]^{2+}$ moiety in C_{2V} symmetry [69]. Those two infrared spectral bands are allocated to$\nu_{asym(O=Mo=O)}$ and $\nu_{sym(O=Mo=O)}$ vibrations, respectively [69-76]. $\nu_{asym(O=Mo=O)}$vibrations are lower than ones of $\nu_{sym(O=Mo=O)}$[77,78]. The presence of acetylacetonate group found in the outer coordination sphere is affirmed using the bands performing around 1552 - 1569 cm^{-1}, and 1467 - 1480 cm^{-1} are appointed to $\nu_{C=O}$ and $\nu_{C=C}$ vibrations [79]. Infrared spectral bands of the macrocyclic complexes exhibit unequal fashion of spectral bands. The asymmetrical and symmetrical N-H stretching vibrations of terminal amino groups disappear because of the binding of those amino groups with a carbonyl group of 1,3-diketones in cyclization methods [79,80].

Table 2. IR spectral bands (ν / cm^{-1}) of ligand and dioxomolybdenum complexes. All spectra had been recorded using KBr within the variety 4000 - 400 cm^{-1}.

Complex	$\nu_{C=N}$	$\nu_{Mo\text{-}N}$	$\nu_{C=O}$ **of acetylacetonate**	$\nu_{C=C}$ **of acetylacetonate**	ν_{asym} (O=Mo=O)	ν_{sym} (O=Mo=O)	ν_{asym} (N-H)	ν_{sym}(N-H)
L	1665s	---	---	---	---	---	3323br	3130br
$[MoO_2(L)](acac)_2$	1604s	508m	1569s	1467m	893s	981s	3433br	3065br
$[MoO_2(ML^1)](acac)_2$	1645m	580m	1555m	1470m	902s	975s		
$[MoO_2(ML^2)](acac)_2$	1640s	591s	1564s	1480s	905m	964s		
$[MoO_2(ML^3)](acac)_2$	1644m	585s	1552m	1475m	910s	965m		
$[MoO_2(ML^4)](acac)_2$	1640s	524m	1560s	1472m	902m	968s		

3.2. ^{1}H NMR spectra.

Spectra ^{1}H NMR of ligand and all prepared MoO_2(VI) complexes were executed in DMSO-D_6. ^{1}H NMR spectrum of produced free ligand proposes signal because of NH_2 at δ (10.20) which is additionally present in $[MoO_2(L)](acac)_2$ at δ (10.12) yet missing in various four macrocyclic complexes $[MoO_2(ML)](acac)_2$ which recommend the cyclization by 1,3-diketones. The ten protons present as multiplets inside the range δ (7.06-7.84) for the isolated ligand and molybdenum complexes. The protons of the aromatic ring appeared by peaks about δ (7.26). ^{1}H NMR spectrum about δ (3.29-3.82) allotted to the CH_2N fragment. The appearance of these chemical shifts might directly result from the arrangement of two types of azomethine that is engaged in the preparation of the macrocyclic complex. The sharp singlet signal situated at δ (2.47) might be because of the water present in DMSO-D_6 sample used.

Table 3. ^{1}HNMR Spectral Data of prepared ligand and dioxomolybdenum complexes (in δ ppm).

Complex	**HC-Ar**	**N-H**	**C-H_3**	**C-H**
L	7.12 10H	10.20 4H	--	--
$[MoO_2(L)](acac)_2$	7.74 10H	10.12 4H	2.46 12H	5.71 2H
$[MoO_2(ML^1)](acac)_2$	7.42 10H	--	2.69 12H	5.58 2H
$[MoO_2(ML^2)](acac)_2$	7.06 10H	--	2.45 12H	5.70 2H
$[MoO_2(ML^3)](acac)_2$	7.84 10H	--	2.70 12H	5.52 2H
$[MoO_2(ML^4)](acac)_2$	7.15 10H	--	2.49 12H	5.58 2H

3.3. UV - Visible spectra.

The Ultraviolet-Vis spectra of tetradentate tetraaza ligand and the dixomolybdenum(VI) complexes had been recorded in ethanol and those spectral bands are measured consistent with the suggested strength energy level scheme [81-83]. The spectra of the dixomolybdenum(VI) complexes with tetradentate ligand are comparable, thereby suggesting a widespread structure for all. In view that Mo(VI) ion has no d- electron, the absorption bands of pure d-d origins do now not appear to be expected to appear. The bands for all complexes may additionally perhaps be appointed as charge transfer transition from nitrogen orbital to a molybdenum metal d-orbital [N(π)→*d*(Mo)]. The Ultraviolet - Vis spectra are homogeneous to rest complexes of dioxomolybdenum(VI) having nitrogen donor atoms. The Ultraviolet - Vis spectra of those complexes are distinguished by strong absorption bands within the UV region at ≈ 292 nm and at ≈ 311 nm appear to be due to intraligand transition and n → π* / π → π* transitions. A fairly intense band that appeared in the vicinity ≈ 380 nm - 395 nm is attributed to N(π)→*d*(Mo). The band due to the transition $^2B_2 \rightarrow {}^2A_1$ ($d_{xy} \rightarrow d_{x2-y2}$) is perhaps covert through the above bands and ought to be appointed for L→M charge-transfer transition among the lowest unoccupied molybdenum d-orbital and highest occupied ligand molecular orbital [84-86]. Ballhausen - Gray energy level diagram has supplied energy level scheme for these complexes. The electronic spectra represent a distorted octahedral configuration for all the complexes [87].

The dioxomolybdenum(VI) complexes are diamagnetic, as pointless to mention for d^0 configuration. Since there is no electron present in the d-orbital, no d-d transitions are decided for those complexes. The molar conductivity (ΛM) values for all dioxomolybdenum(VI) complexes in DMF at ca. 10^{-3} M endorse 1:1 sort electrolytes. The molar conductance values of those complexes lie between the ranges of 100 - 115 Ω^{-1} cm^2 mol^{-1}. In final, the on top molar conductance values aid the tentative structures of dioxomolybdenum(VI) complexes of the type (I) and macrocyclic complexes of type (II) as shown in the schemes.

3.5. Thermogravimetric analyses.

The thermogravimetric investigation of $[MoO_2(L)](acac)_2$ complex has been governed in the temperature range 50 - 600 °C with a 10 °C min^{-1} temperature in-between. No transparent decomposition was remarked beneath 145 °C (Figure 1). The thermograph of the $[MoO_2(L)](acac)_2$ complex is shown in Figure 1. The $[MoO_2(L)](acac)_2$ complex undergoes decay in two levels: (a) first degree of decay (145 - 250 °C) is because of the loss of ligand (mass loss obs. 45 %, calcd. 48 %) (b) in the second degree, the second successive weight loss of at raised temperature (300 - 450 °C) giving a mass loss over 42 % against calculated mass loss of 48 %.

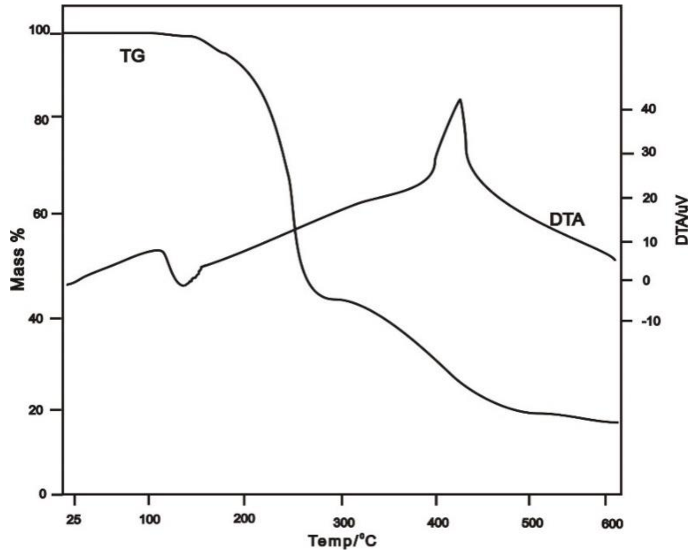


Figure 1. TG and DTA thermographs of $[MoO_2(L)](acac)_2$.

At final, a residue obtained generally compares to MoO_3 (obs. residual mass = 14.50 %, calcd. = 15.75 %) were left after 450 °C. Two vertexes had been gotten in the DTA curve. The first vertex is endothermic decay due to the melting of the complex (145 °C). The second vertex is because of the exothermic effect. Close to this exothermic vertex in the DTA curve, an exothermic hump was also observed at 435 °C, which may also be due to the exothermic decay of the leftover mass in the second step.

3.6. Antibacterial activity.

The consequences of the antimicrobial activity of the prepared dioxomolybdenum(VI) complexes are given in Table 3. The synthesized dioxomolybdenum(VI) complexes had been attempted towards *Staphylococcus aureus, Bacillus subtilis, Enterobacter aerogenes*,and *Salmonella typhi*. The improvement in the antibacterial interest of dioxomolybdenum(VI) complexes can be clarified on the idea of the chelation hypothesis [79,80]. The reference material is doxycycline remedy. Nearly all the complexes specified low to modest activity towards *S. aureus* and *S. typhi*.

Table 4. Antibacterial activities ofmacrocyclic complexes of dioxomolybdenum(VI).

Complex	***Staphylococcus aureus***	***Enterobacter aerogenes***	***Salmonella typhi***	***Bacillus subtilis***	***Doxycycline***
[MoO2(L)](acac)2	17	17	19	20	26
[MoO2(ML1)](acac)2	14	20	17	22	23
[MoO2(ML2)](acac)2	14	17	16	20	24
[MoO2(ML3)](acac)2	15	19	19	--	25
[MoO2(ML4)](acac)2	17	19	--	22	25

4. Conclusions

As the crystals of complexes were not isolated, we cannot put forward the crystal structures of the respective complexes. From the above elemental and spectral studies, we may also advise that all the complexes synthesized can be represented as $[MoO_2(L)](acac)_2$ and $[MoO_2(ML^1)](acac)_2$. The antibacterial activity has been carried out on these complexes, which screen that the complexes are biologically active.The present study manifest primary synthetic paths to get new dioxomolybdenum(VI) with Schiff base. The applied spectroscopic methods have affirmed the Schiff base condensation of di-2-furanylethanedione, a flexible chelating agent with two responsive carbonyl groups with diamines, and their cyclizations 1,3-diketones present process formation of macrocyclic products to assure managed geometry around MoO_2(VI) center. The shape around Mo is distorted octahedral geometry. The kinetic template impact of dioxomolybdenum(VI) cation assumes a considerable function in the condensation of Schiff base using di-2-furanylethanedione and diamines in ethanol medium. Synthesized Schiff bases behave as tetradentate ligands by binding to the metal ion along with the azomethine nitrogen atoms. The existence of one metal ion per ligand molecule is declared by analytical data. The mononuclear six coordination of all the prepared *cis*-dioxomolybdenum(VI) complexes and the six-coordinate distorted octahedral shape have been suggested for these complexes.

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Conflicts of Interest

The authors declare no conflict of interest.

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