

Thiophenyl Azanylidene Pentadamine Molybdenum Compounds and Their Antibacterial Effects

Siddharam ¹, Nidhi ¹, Amit Kumar Gautam ¹, Devendra Pratap Rao ^{1,*}

¹ Department of Chemistry, Coordination Chemistry Laboratory, Dayanand Anglo-Vedic (PG) College, Chhatrapati Shahu Ji Maharaj University, Kanpur-208001, Uttar Pradesh, India

* Correspondence: devendraprataprao_kn03@csjmu.ac.in;

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Abstract: One thiophenyl azanylidene pentadamine Schiff base (TAPSB), one of their molybdenum parent compounds [MoO₂(TAPSB)], and four macrocyclic compounds [MoO₂(MTAPSB)] were successfully synthesized. Their structures were analyzed comprehensively using spectroscopic techniques. This paper explores the interaction between β-diketones and [MoO₂(TAPSB)]. In totality, five MoO₂(VI) compounds were investigated by virtue of modern-day UV-Vis, magnetic, IR, and NMR characterization protocols. In these compounds, coordination of two oxo (O) and four nitrogen (N) atoms forms the octahedral geometry around molybdenum. Entirely produced compounds presented effectiveness in response to *S. aureus* and *S. typhi*. The antibacterial job is a good manifestation of the chelation hypothesis.

Keywords: antibacterial activity; Mo(VI); thenil; β-diketones; quinoxaline; chelation.

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1. Introduction

A minimum of nine atoms is present in a macrocyclic compound. Among these nine atoms, a minimum of three donor sites, including all heteroatoms, are present. Stupendous growth has been observed in macrocyclic compounds over the past five decades [1-5]. Nitrogen-containing ligands and related compounds are crucial in coordination chemistry. Many research investigations that study main-group complexes from biological perspectives and promote wide-ranging applications have been published [6-11]. Using metal ions, a main-group Schiff base containing vitamin B6 catalyzes transamination reactions [12-16]. Schiff base compounds exhibit anti-inflammatory, anticancer, antifungal, and antiviral properties [12–20].

Schiff bases in the bioinorganic sector can be used to simulate the metal sites found in metalloproteins in consonance with enzymes [21, 22]. Several compounds of Schiff bases show more anticancer activity for that specific metal complex than the isolated ligand [23–32]. They also find applications in electronics, memory devices, and chemical sensors.

Ligands with various coordination numbers and geometries, with penultimate density, in transition-metal compounds have been studied [33,34]. Multiple oxidation states have made molybdenum an appropriate choice for the present study, with coordination numbers ranging from 4 to 8 [32,33]. Mo compounds equipped with other ligands containing heteroatoms. These Schiff base structures may be thoroughly investigated in both conditions, viz., homogeneous

and heterogeneous. A wide spectrum of biological applications is envisioned for MoO₂ compounds with multiidentate ligands [35-41]. The role of the [MoO₄]²⁻ ion is smartly delineated as a template for oxygen transfer. The complete oxidation of a number of redox enzymes is known [42-46]. Active sites of these enzymes remain equipped with the cis-dioxomolybdenum moiety [47-49].

Molybdenum is a quintessential bio-metal across a wide spectrum of pathogens and microorganisms, as suggested by numerous studies [50,51]. Many studies have focused on unraveling the biological importance and the catalytic properties of Mo(VI) in coordination chemistry across different species [52-55]. Oxomolybdoenzymes are set up by molybdenum molecules [56-58]. One of the bona fide applications of Thenil could be found in the capacity of a chelating agent with an array of applications citing to its activity owing to the main functional groups, carbonyl groups, diamines, and thenil, which corresponds to Schiff base condensation.

Reactions of diamine with thenil counterpart synthesized thenil's denticity ligands, while the synthesis of 1,3-diketones was impacted by the presence of metal carriers. The structures were characterized using advanced instrumentation, e.g., NMR, FTIR, and UV-visible spectroscopy, in addition to molar conductivity measurements, to ascertain their authenticity and purity.

2. Materials and Methods

2.1. Materials.

Reagent-grade thenil and 1,3-pentanediamine were used for the preparation of the Schiff base (TAPSB). Additionally, we obtained Mo(acac)₂, 1,3-pentanediamine, thenil, and 1,3-diketones from Aldrich.

2.2. Analytical methods.

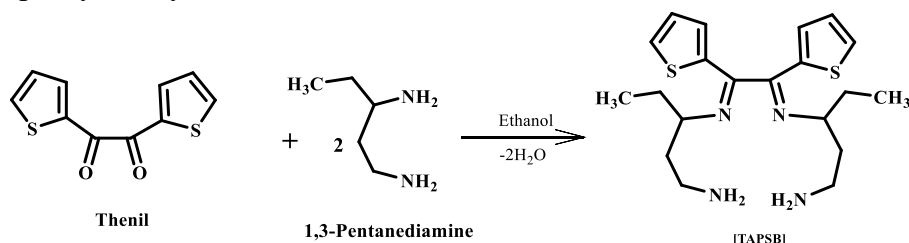
The nitrogen content was determined by the official Kjeldahl method. The molybdenum content of the synthesized analogues was measured by the gravimetric method [59], and sulfur content was determined as barium sulfate by standard gravimetric analysis [60]. Melting points were analyzed by virtue of applying vitriol baths in addition. A Labinda spectrophotometer was used to obtain UV spectra in the choice of solvent, ethanol. Similarly, Infrared spectra of the MoO₂(VI) compound were obtained using the ATR facility at the UPTTI, Kanpur. ¹H NMR spectrum of MoO₂(VI) was drawn by the virtue of JEOL's JMM ECS-400 spectrometer. TG recording was done at IITK, India. The thermal behavior of the [MoO₂(TAPSB)] compound was examined at 10°C/min over 50-900°C under nitrogen conditioning. ESI-MS analysis of the synthesized analogues was performed at the instrumentation facility using Agilent mass spectrometers at IIT Kanpur.

2.3. Synthesis of thiophenyl azanyllidene pentadamine Schiff base (TAPSB).

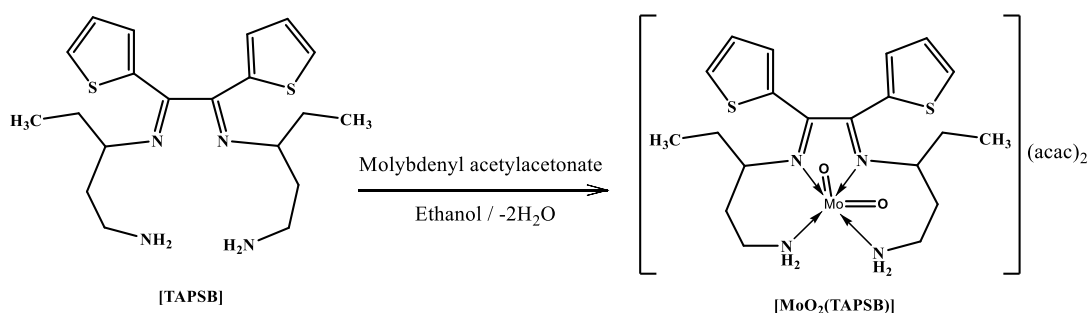
The Schiff base was synthesized by mixing thenil (5 mmol) and 1,3-pentanediamine (10 mmol) in ethanol, followed by refluxing the mixture for 1 hour, yielding a pale yellow solution. The pale yellow product was scored by removing excess solvent under vacuum at room temperature, as explained in Scheme 1. The yield of the product was good, around 70%, and was determined by repeatedly washing the complex with methanol.

2.4. *In-situ* preparation of $[MoO_2(TAPSB)]$ compound with TAPSB.

Schemes 1 and 2 illustrate the synthesis of $[MoO_2(TAPSB)]$. 4 mmol of $MoO_2(acac)_2$ dissolved in MeOH (30 mL) was added to a solution of 4 mmol of thenil and 8 mmol of 1,3-pentanediamine in ethanol (40 mL). After six hours of a little reflux in the reaction mixture, the solution turns yellow (Scheme 2). Following filtering, the solid product was cleaned with ethanol and then vacuum-isolated using silica gel. The TLC method was applied to verify the compound's purity. The yield of $[MoO_2(TAPSB)]$ was 60 %.



Scheme 1. Preparation of ligand (TAPSB).

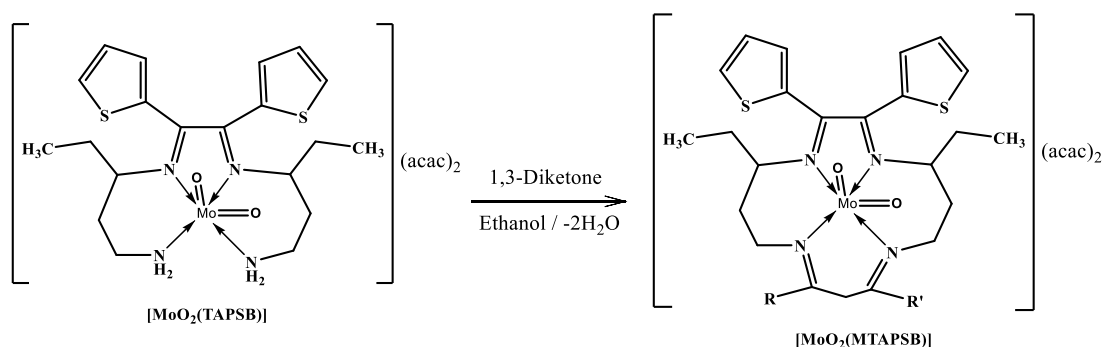


Scheme 2. Preparation of $[MoO_2(TAPSB)]$.

2.5. *In-situ* preparation of $[MoO_2(MTAPSB)]$ compound with $[MoO_2(TAPSB)]$.

Added $MoO_2(acac)_2$ (5 mmol) into ethanol drop-wise. Then, an ethanolic solution of thenil (5 mmol) and 1,3-pentanediamine (10 mmol) was added to the molybdenum salt solution. The R.M. has been refluxed for 3 hrs mildly, and the solution becomes a dirty yellow colour (Scheme 3). The product was washed with methanol. The product was filtered after vacuum isolation using silica gel. The TLC method was applied to verify the compound's purity. The yield of $[MoO_2(MTAPSB)]$ increased to 60%. $[M+H]^+$ analysis yielded a molecular ion peak at 277.09 (m/z). Scheme 3 shows the preparation of $[MoO_2(MTAPSB)]$.

In Figures 1 and 2, Schiff base ligand (TAPSB) and $[MoO_2(TAPSB)]$ are shown as 3D structures.



Scheme 3. Synthetic route to $[MoO_2(MTAPSB)]$. MTAPSB1: R' = CH₃, R = CH₃; MTAPSB2: R' = CH₃, R = C₆H₅; MTAPSB3: R' = CF₃, R = C₄H₃S; MTAPSB4: R' and R = C₆H₅

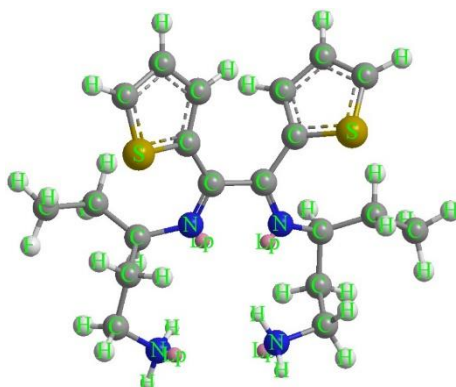


Figure 1. 3D structure of the TAPSB molecule generated using Chem3D software.

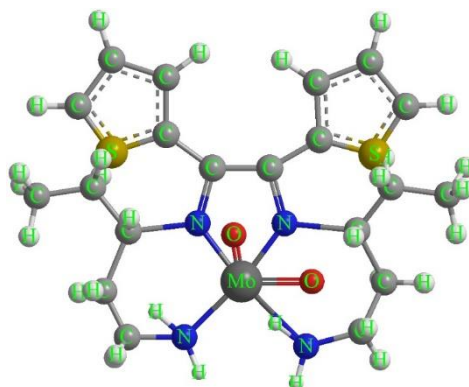


Figure 2. [MoO₂(TAPSB)] 3D structure.

A solid product was formed from 1,3-diketones and [MoO₂(TAPSB)] in EtOH after 3 hours. MoO₂(MTAPSB), a yellow pigment with a solid, macrocyclic nature, was further subjected to TLC to address purity concerns. The nature is corroborated by the data shown in Table 1.

Table 1. CHN data.

Compound	Yield (%)	Empirical formula	Formula weight	m.p. (°C)	% Calcd. / (found)				
					C	S	N	Mo	H
TAPSB	55	C ₂₀ H ₃₀ N ₄ S ₂	390.61	120	61.50 (61.20)	16.42 (16.0)	14.34 (14.21)	--	7.74 (7.51)
[MoO ₂ (TAPSB)]	40	C ₃₀ H ₄₄ MoN ₄ S ₂ O ₆	716.77	140	50.27 (50.15)	8.94 (8.82)	7.81 (7.72)	13.38 (13.20)	6.18 (6.10)
[MoO ₂ (MTAPSB ¹)]	45	C ₃₅ H ₄₈ MoN ₄ S ₂ O ₆	780.86	130	53.83 (53.50)	8.21 (8.15)	7.17 (7.05)	12.29 (12.21)	6.19 (6.15)
[MoO ₂ (MTAPSB ²)]	48	C ₄₀ H ₅₀ MoN ₄ S ₂ O ₆	842.93	135	57.00 (56.92)	7.60 (7.55)	6.64 (6.54)	11.38 (11.30)	5.97 (5.85)
[MoO ₂ (MTAPSB ³)]	50	C ₃₈ H ₄₅ MoN ₄ S ₃ O ₆ F ₃	902.93	130	50.54 (50.45)	10.65 (10.60)	6.20 (6.10)	10.65 (10.57)	5.02 (4.95)
[MoO ₂ (MTAPSB ⁴)]	45	C ₄₅ H ₅₂ MoN ₄ S ₂ O ₆	905.00	132	59.72 (59.65)	10.60 (10.55)	6.19 (6.15)	10.61 (10.54)	5.79 (5.60)

TAPSB = Thiophenyl Azanyllidene Pentadiazine Schiff base; [MoO₂(MTAPSB¹)] = product with TAPSB and 2,4-pentanedione; [MoO₂(MTAPSB²)] = product with TAPSB and Benzoylacetone; [MoO₂(MTAPSB³)] = product with TAPSB and 2-Thenyltrifluoroacetone; [MoO₂(MTAPSB⁴)] = product with TAPSB and Dibenzoylmethane 1:1 metal: ligand complexes.

2.6. Antibacterial activity assay.

The synthesized compound was tested against a wide array of bacterial strains to assess its antibacterial efficacy using an agar-well diffusion protocol, with doxycycline and trademark antibiotics as reference drugs [61-63]. Bacterial suspensions were precisely diluted to 3x10⁵

CFU/mL. The standardized suspensions were optimized to expand agar. An investigation was carried out in a 1% DMSO solution containing 300 g L⁻¹ of the produced compound, and all samples were poured into each well accordingly. The remaining wells were treated with 0.05% DMSO and 0.5% doxycycline, and growth inhibition was assessed after 35 hours at 35°C.

3. Results and Discussion

3.1. Infrared spectra.

Schematic representation reflects the in-situ synthesis of MoO₂(VI) having components in the capacity of diamine, thenil and molybdenum salt, in a 2:1:1 ratio with aq. EtOH solution and ultimately leading to a unique product. IR spectra of the compounds showed several bands (Figure 3), as explained in Table 2. Decreased νC=N absorption frequencies of azomethine N-atoms coordinated to Mo macrocyclic species were facilitated in the spectra [63-67]. An isolated ligand >C=N absorbances at 1633-1675 cm⁻¹ were observed [64-66], along with non-ligand vibration (Mo-N) at 519-575 cm⁻¹ as a possible explanation for the same [68]. A strong >C=N bands at around 1660 cm⁻¹ was scrutinized to corroborate that thenil is a robust ligand for diamines [69,70]. Numerous rings along with an array of C-H vibrations were observed in the IR spectra of MoO₂(VI) compounds and isolated ligands, entailing asym and sym(N-H) bands at 3430 and 3072 cm⁻¹, respectively. Identical structures of [MoO₂(TAPSB)] and [MoO₂(MTAPSB)] were validated by the non-appearance of the NH group [71]. The d-orbital is most frequently used for bonding in dioxomolybdenum(VI) compounds, yielding preferentially cis-dioxo groups. Due to C_{2v} symmetry, cis-[MoO₂]²⁺ exhibits both symmetric and asymmetric stretching at 944-975 and 896-955 cm⁻¹ in dioxomolybdenum(VI) compounds [72]. The ν_{asym} and ν_{sym}(O=Mo=O) dioxomolybdenum frequencies are linked to twin IR bands at the corresponding wavelengths [72-79].

Table 2. IR bands of compounds.

Compound	ν of							
	C=N	Mo-N	C=O (acac)	C=C (acac)	asym (O=Mo=O)	sym (O=Mo=O)	asym (N-H)	Ym (N-H)
TAPSB	1675	---	---	---	---	---	3325	3084
[MoO ₂ (TAPSB)]	1633	519	1577	1406	955	944	3430	3072
[MoO ₂ (MTAPSB ¹)]	1670	575	1565	1464	900	975		
[MoO ₂ (MTAPSB ²)]	1665	530	1562	1470	905	970		
[MoO ₂ (MTAPSB ³)]	1642	560	1560	1468	896	968		
[MoO ₂ (MTAPSB ⁴)]	1645	555	1568	1473	899	965		

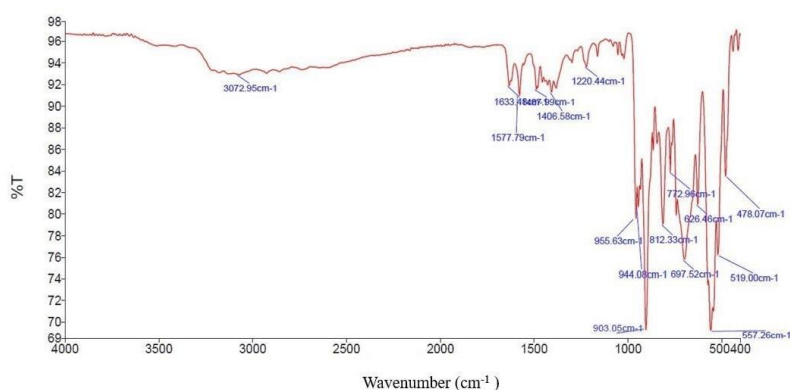


Figure 3. IR spectra of the [MoO₂(TAPSB)] compound.

The $\nu_{\text{sym}}(\text{O}=\text{Mo}=\text{O})$ was examined at a lower energy than the $\nu_{\text{sym}}(\text{O}=\text{Mo}=\text{O})$ [80,81]. Both the C=C and C=O frequencies came at $1562\text{-}1577\text{ cm}^{-1}$ and $1406\text{-}1473\text{ cm}^{-1}$, respectively, in the outer coordination sphere [82]. An equal arrangement of IR bands is displayed by the macrocyclic substances. Both the vibrations of the amino group tagged terminally vanish when 1,3-diketones are cyclized by adding -C=O groups to the -NH₂ group [82, 83].

3.2. ¹H NMR spectra.

Ligand and MoO₂(VI) complexes were rationalized for ¹H NMR in DMSO-D₆. The free ligand (TAPSB) shows an NH₂ signal at δ 1.50, which shifts to δ 7.20 in [MoO₂(TAPSB)], confirming coordination. All protons denoting ligand were observed in the range δ (7.53-0.89) encoding multiplets for isolated ligands and molybdenum compounds. Aromatic protons were observed at δ 7.40. The most striking explanation for the signals may be assigned to two types of C=N, which facilitated the preparation of the macrocyclic complex during the synthesis.

¹H NMR in DMSO-D₆: TAPSB: N-H (δ = 1.50, 4H), HC-diamine (δ = 2.14, 2H), H₃C-diamine (δ = 0.89, 6H), H₂C-diamine (δ = 2.63, 4H), HC-thiophene (δ = 7.53, 2H; 7.15, 2H; 7.01, 2H); [MoO₂(TAPSB)]: N-H (δ = 7.20, 4H), HC-diamine (δ = 1.3, 2H), H₃C-diamine (δ = 0.89, 6H), H₂C-diamine (δ = 2.19, 4H; 3.36, 4H), HC-thiophene (δ = 7.53, 2H; 7.15, 2H; 7.01, 2H); [MoO₂(MTAPSB¹)]: HC-diamine (δ = 1.3, 2H), H₃C-diamine (δ = 0.89, 6H), H₂C-diamine (δ = 1.40, 8H), HC-thiophene (δ = 7.53, 2H; 7.15, 2H; 7.01, 2H); [MoO₂(MTAPSB²)]: HC-diamine (δ = 1.3, 2H), H₃C-diamine (δ = 0.89, 6H), H₂C-diamine (δ = 1.40, 8H), HC-thiophene (δ = 7.53, 2H; 7.15, 2H; 7.01, 2H), HC-Ar (δ = 7.40, 5H); [MoO₂(MTAPSB³)]: HC-diamine (δ = 1.3, 2H), H₃C-diamine (δ = 0.89, 6H), H₂C-diamine (δ = 1.40, 8H), HC-thiophene (δ = 7.53, 2H; 7.15, 2H; 7.01, 2H), HC-thenoyl (δ = 7.53, 1H; 7.15, 1H; 7.01, 1H); [MoO₂(MTAPSB⁴)]: HC-diamine (δ = 1.3, 2H), H₃C-diamine (δ = 0.89, 6H), H₂C-diamine (δ = 1.40, 8H), HC-thiophene (δ = 7.53, 2H; 7.15, 2H; 7.01, 2H), HC-Ar (δ = 7.40, 10H).

3.3. UV - Visible spectra.

Dioxomolybdenum(VI) compounds and ligand were found consistent, implying a strong energy level in ethanol solvent [84-87]. The spectra of molybdenum(VI) compounds and TAPSB are comparable. The Mo(VI) ion is no longer anticipated to exhibit a pure d-d transition since it lacks a d-electron. It is also possible to think of all prepared Mo compounds as electronic transitions [N(π) \rightarrow d(Mo)] between d-orbitals of Mo and N. The UV-VIS spectra of a dioxomolybdenum(VI) molecule with N- atoms are uniform. Transitions like intraligand and $n\rightarrow\pi^*$ / $\pi\rightarrow\pi^*$ may be responsible for the strong absorption bands seen at 290 nm and 315 nm in those compounds' UV-Vis spectra, and explain a conceivable reason for N(π) \rightarrow d(Mo) in the region of 380–395 nm for the assigned band. Other bands, which occur by virtue of transitions ${}^2B_2\rightarrow{}^2A_1$ ($d_{xy}\rightarrow d_{x^2-y^2}$), are well tolerated for mediating charge transfer between the rotational energy levels of HOMO and LUMO. Illustrative Ballhausen-Gray diagrams detailing energy levels are provided for the compounds, emphasizing an octahedral distortion in their electronic spectra [88-90].

3.4. Magnetic properties.

With the d⁰ configuration, dioxo molybdenum (VI) did not express any magnetic property. No d-d transitions calculations were offered for the synthesized analogues, as these electrons were found to be missing in the d-orbitals. Ratio 1:1 sort electrolytes are supported

by the molar conductivities (Λ_M) of every Mo compound in DMF at about 10^{-3} M. The synthesized analogues were found exhibiting Λ_M values of 97 and $115 \Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$. Values of molar conductance were approximated for both $[\text{MoO}_2(\text{TAPSB})]$ and $[\text{MoO}_2(\text{MTAPSB})]$ compounds, as shown in the schemes.

3.5. ESI mass spectrum.

Mass spectrum for the synthesized compound $[\text{MoO}_2(\text{TAPSB})]$ is systematically interpreted in Figure 4. $m/z = 277$ of $[\text{M}+\text{H}]^+$ ion peak appeared in the given spectrum, citing to involvement of varied isotopes entailing nitrogen and sulphur atoms, and the synthesized structure was found to be quite stable. In the spectrum, no other relatively intense peaks appeared to be explained.

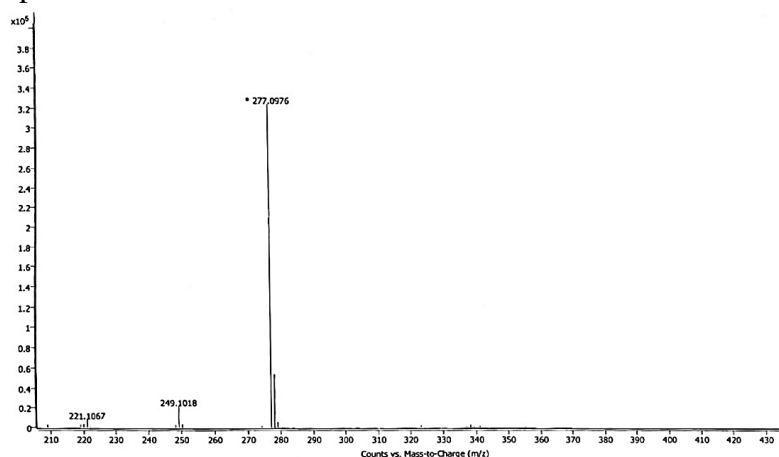


Figure 4. The $[\text{MoO}_2(\text{TAPSB})]$ compound's mass spectrum.

3.5. Antibacterial activity.

Table 3 explained the antimicrobial efficacy of the dioxomolybdenum (VI) complex. Screened against a broad spectrum of bacteria, e.g., *B. subtilis*, *S. aureus*, *E. aerogenes*, and *S. typhi* with their rational activities. An amicable explanation for the stupendous antibacterial efficacy of Mo(VI) compounds could be traced in the chelation presumption [82,83]. Against *B. subtilis* and *S. typhi*, modest activity was observed for all compounds.

With reference to the positive control, the percentage inhibition effect was enforced employing Eq. 1.

$$\% \text{ inhibition effect} = \frac{\text{extract inhibition halo diameter}}{\text{positive control inhibition halo diameter}} \times 100 \quad (1)$$

Table 3. Antibacterial activities of compounds

Compound	<i>S. aureus</i>	<i>E. aerogenes</i>	<i>S. typhi</i>	<i>B. subtilis</i>	<i>Doxycycline</i>
$[\text{MoO}_2(\text{TAPSB})]$	20	21	19	15	25
$[\text{MoO}_2(\text{MTAPSB}^1)]$	17	20	16	16	24
$[\text{MoO}_2(\text{MTAPSB}^2)]$	19	19	17	15	24
$[\text{MoO}_2(\text{MTAPSB}^3)]$	19	20	16	17	26
$[\text{MoO}_2(\text{MTAPSB}^4)]$	18	19	17	16	24

$300 \mu\text{g mL}^{-1}$ of material was dissolved in DMSO in well cups and wells following the protocol to assess the compound efficacy. 19-22, 15-18, range for activity spanning good, moderate, and 23-26 range strong ambit was found in the following range with reference to doxycycline to access the inhibitory activity of the synthesized analogues (mm).

InChI

InChI=1S/C20H30N4S2.Mo.2O/c1-3-15(9-11-21)23-19(17-7-5-13-25-17)20(18-8-6-14-26-18)24-16(4-2)10-12-22;;;/h5-8,13-16H,3-4,9-12,21-22H2,1-2H3;;;/b23-19+,24-20+;;;/t15-,16+;;;

[MoO₂(TAPSB)] InChI key

VAZMQSVSZPJEOC-GPTKBHRNSA-N

[MoO₂(TAPSB)] SMILES

CCC([N+]=C(C(C2=CC=CS2)=[N+])3C(CC[NH2+])4)CC)C5=CC=CS5)CC[NH2+][Mo-4]134(=O)=O

LogP

0

4. Conclusions

The crystal structure for the corresponding compound is not available. Otherwise, the structure [MoO₂(TAPSB)] or [MoO₂(MTAPSB)] was corroborated based on the inputs from a spectrum of sources in the ambit of elemental and spectral studies, followed by the spectroscopical investigations. The antibacterial efficacy of the compounds was evaluated against a wide array of bacterial strains. The present study demonstrates dioxin derivatives produced from the TAPSB of Mo(VI). The property of a flexible chelator was supported by the presence of two carbonyl groups in thenil. After reacting with 1,3-diketones, they produce macrocyclic compounds that regulate the geometrical shape of MoO₂(VI) centers. The octahedron surrounding Mo is distorted. Condensation of Schiff bases involves dioxomolybdenum(VI) cation in an ethanol medium using thenil and diamines. Tetradentate bonds are created between the metal ions and the azomethine nitrogen atoms in manufactured TAPSB. The synthesized compounds are proposed to have distorted octahedral, six-coordinate, mononuclear geometries with promising antibacterial activity.

Author Contributions

Software, S. and A.K.G.; investigation, S.; methodology, N.; visualization, N.; validation, A.K.G.; data curation, A.K.G.; writing—review and editing, A.K.G.; formal analysis, D.P.R.; resources, D.P.R.; writing—original draft preparation, D.P.R.; supervision, D.P.R.; project administration, D.P.R.; funding acquisition, D.P.R. All authors have read and agreed to the published version of the manuscript.

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Informed Consent Statement

Not applicable.

Data Availability Statement

Data supporting the findings of this study are available upon reasonable request from the corresponding author.

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Conflicts of Interest

The authors declare no conflict of interest.

Role of Funders

The funders had no role in the design of the study, in the collection, analysis, or interpretation of data, in the writing of the manuscript, or in the decision to publish the results.

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