



Therapeutic Studies of the Dioxomolybdenum (VI) Compounds of Schiff Base

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ABSTRACT

One thiophenyl azanylidene binaphthyl-2,2'-diamine Schiff base (TABSB), one of their molybdenum parent compounds (TABSBMo), and four macrocyclic compounds (TABSBMoβs) were successfully synthesized. Their structures were analyzed comprehensively using spectroscopic techniques. This paper entails a nexus of research of the interaction between β-diketones and (TABSBMo). This article describes the characterization of five MoO₂(VI) compounds using UV-Vis, IR, NMR, and TG. Molybdenum metal will be in possession of a coordination number of six. There is a distorted octahedral arrangement geometry in each of MoO₂(VI)'s five compounds. In these compounds, the molybdenum octahedral geometry is completed by two oxidized O-atoms with four N-atoms. When it came to *S. aureus* and *S. typhi*, all of the synthesized compounds showed a mild level of effectiveness. The antibacterial job is smart manifestation pertaining to chelation hypothesis.

Keywords: Antibacterial activity, Thenil, β-diketones, macrocyclic compounds, 1,1'-Binaphthyl-2,2'-diamine and dioxomolybdenum (VI).

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. Macrocyclic substances have advanced considerably over the last fifty years¹⁻⁵. 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 uses have been published⁶⁻¹¹. Using metal ions, a main group Schiff base containing vitamin B6 catalyzes transamination methods^{15,16}.

Schiff base compounds have anti-inflammatory together as anti-cancer properties and to a greater extent with corresponding antifungals well as antiviral ones¹²⁻³⁰.

Schiff bases in the bioinorganic sector can be used to simulate the metal sites found in metalloproteins in consonance with enzymes^{31,32}. They also still find consumer electronics, memory storage devices, and chemical interactions fascinating.

The coordination number and arrangement of extreme density ligands in transition metal



complexes vary^{33,34}. Molybdenum can be flexible in this regard due to its various oxidation states. Moreover, a coordination number can be between 4 and 8^{32,33}. By connecting molybdenum compounds along with ligands that contain heteroatoms (N, O, and S), it is possible to construct Schiff base structures for both homogeneous and heterogeneous processes³⁷⁻⁴¹. Since dioxomolybdenum(VI) molecules have multidentate ligands, they can be used in a variety of biological applications. In an aqueous solution, $[\text{MoO}_4]^{2-}$ is easily obtained as Mo(VI). The pH and concentration of the solution can be used to calculate the $[\text{MoO}_4]^{2-}$ ion concentration. The role of the $[\text{MoO}_4]^{2-}$ ion is smartly delineated as a template for oxygen transfer. The molybdenum oxotransferase mechanism has been demonstrated to be adversely influenced by their properties of oxygen transport⁴²⁻⁴⁶. The complete oxidation of a number of redox enzymes is known⁴⁷⁻⁴⁹. In these situations, their active sites are linked to a cis-dioxomolybdenum moiety.

For pathogenic bacteria that are present in vegetation, animals, and humans, the sole transition metal is molybdenum that is regarded as a biometal that is necessary^{50,51}. Research on Mo(VI) coordination chemistry is ongoing due to its biological and catalytic characteristics⁵²⁻⁵⁵. The physiological activities of oxomolybdoenzymes are established by a molybdenum molecule⁵⁶⁻⁵⁸. Because of its versatility, thenil may be helpful as a chelating agent. The reactive carbonyl groups in thenil may cause Schiff base condensation with diamines. In the synthesis of macrocyclic ligands, di-2-furanylenedione is a crucial component.

Thenil contains denticity ligands that, in some circumstances, can be produced via the interaction of thenil with diamine. The produced chemical is cyclized with 1,3-diketones through the impact of a metal template. Molar conductivity investigations, elemental analysis, UV-Vis, TGA, IR, and NMR spectroscopy have all been used to establish their putative structures and prepare and describe them.

EXPERIMENTAL

Materials

Schiff bases (ligands) and compounds

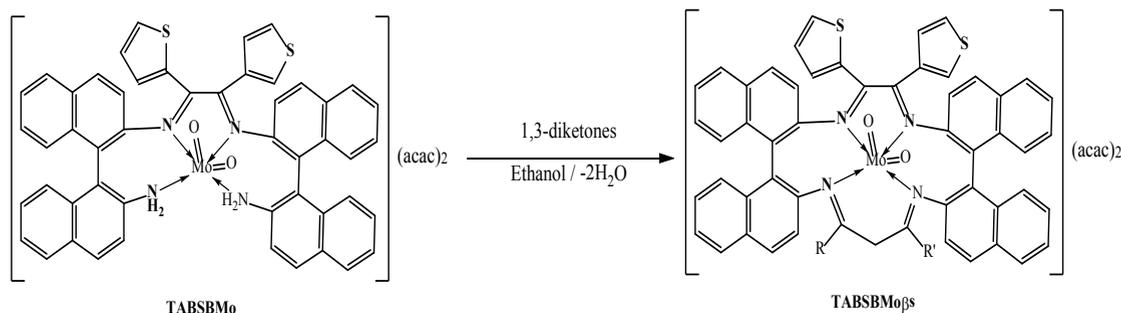
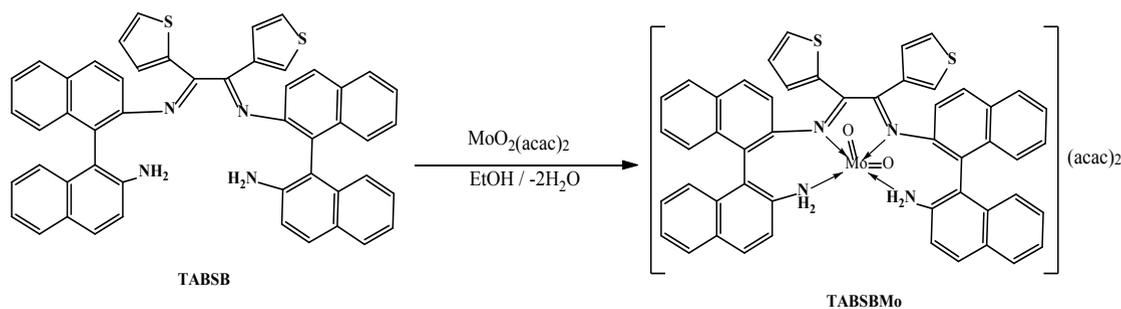
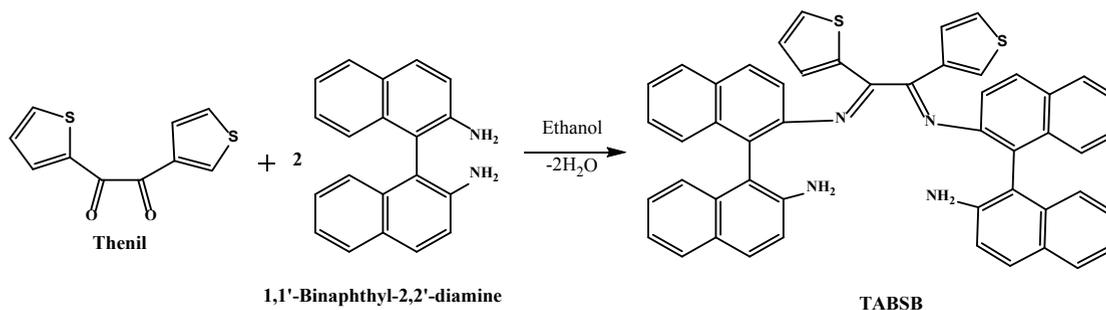
were created by chemists sourced from company resources employing chemicals of reagent grade. The 1,3-diketones were derived from Aldrich without undergoing the same refining as molybdenyl acetylacetonate, 1,1'-Binaphthyl-2,2'-diamine, and thenil.

Physical Measurements and Analytical Methods

CHN analyzers were utilized to look at C, H, and N in the compounds at significant CRFs in Arunachal Pradesh, India, such as NERIST, Nirjuli, and Itanagar. Kjeldahl's method is adopted to measure nitrogen in synthetic substances. Following the compound's disintegration, the gravimetric method was employed to estimate molybdenum⁵⁹. Sulfur estimation advanced with the development of barium sulfate as a sulfur estimation technique⁶⁰. Compound's electronic absorption spectra were manifested using ethanol to the facility of UV spectrophotometer (Labinda) at Uttar Pradesh Textile Technical Institute, Kanpur, U.P., India. Infra-red spectra of $\text{MoO}_2(\text{VI})$ molecules in potassium bromide were practiced by virtue of spectrometers corresponding to Perkin-Elmer Spectrum with models of 10.03.06 and 18.03.06 at IIK. JEOL's JMM ECS-400 was utilized to furnish the proton NMR spectra of $\text{MoO}_2(\text{VI})$. TG recording was done at IITK, India. The thermal behavior of the TABSBMo compound was examined at 10°C per minute over 50-900 °C in a nitrogen conditioning.

Condensation of thenil and 1,1'-Binaphthyl-2,2'-diamine with 1,3-diketones yields molybdenum (VI) compounds with ligands

Schemes 1, 2, and 3 illustrate the synthesis of TABSBMo β s. Thenil (5 mmol, 0.9506 g) and 1,1'-Binaphthyl-2,2'-diamine (10 mmol, 2.0106 g) were added to 60 mL of ethanol after molybdenyl acetylacetonate (5 mmol, 1.6307 g) was added dropwise. After three hours of a little reflux in the reaction mixture, the solution turns brown. 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. Thus, the yield of TABSB was 48%. Comparative 3D framework of Schiff base ligand TABSB along with the fountainhead TABSBMo motif is depicted in Figures 1 and 2.



Where TABSB = thenil + 1,1'-Binaphthyl-2,2'-diamine; TABSBMoβs = Synthesis of macrocyclic ligands by reaction of 1,3-diketones with TABSB in occupation of Mo₂(VI) cation.

R and R' = CH₃ for TABSBMoβs₁; R = C₆H₅, R' = CH₃ for TABSBMoβs₂; R = C₄H₉S, R' = CF₃ for TABSBMoβs₃; R and R' = C₆H₅ for TABSBMoβs₄

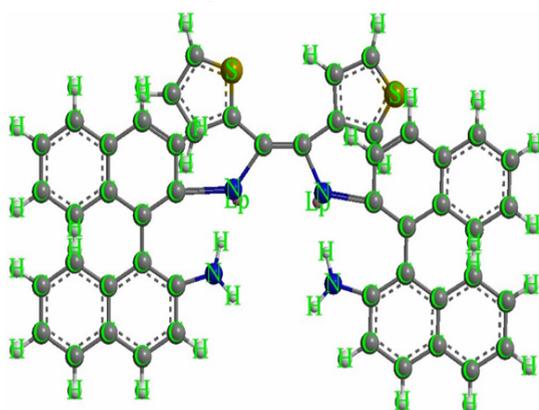


Fig. 1. TABSB3D structure

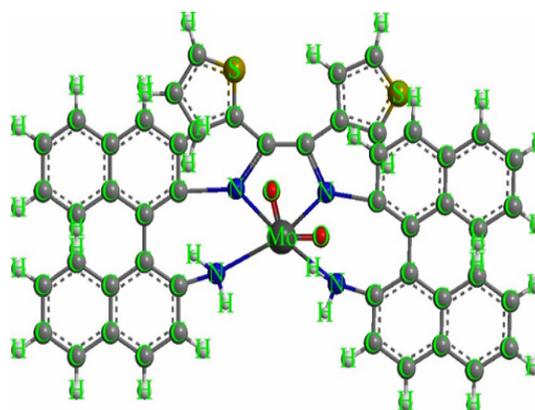


Fig. 2. TABSBMo3D structure

Reaction of 1,3-diketones to TABSBMo's alcoholic solution leads to a consistent reaction lasting for three hours. Solid macrocyclic compounds (type B), yellowish in texture, that we produced here were

1,3-diketones such as (a) 2-Thenoyltrifluoroacetone (b) dibenzoylmethane (c) 2,4-pentanedione, or (d) benzoylacetone. To confirm that macrocyclic compounds are pure, a TLC test is conducted.

Table 1: CHN data

Compound	Formula	F.W.	Yield(%)	m.p.(°C)	%of Element, Calcd./(found)				
					C	H	N	Mo	S
TABS	C ₅₀ H ₃₄ N ₄ S ₂	754.97	60	118	79.55(78.95)	4.54(4.50)	7.42(7.35)	--	8.49(8.40)
TABS	C ₆₀ H ₄₈ N ₄ MoO ₆ S ₂	1081.14	50	130	66.65(66.60)	4.47(4.40)	5.18(5.11)	8.87(8.82)	5.93(5.80)
TABS	C ₆₅ H ₅₂ N ₄ MoO ₆ S ₂	1145.22	48	132	68.17(68.13)	4.57(4.50)	4.89(4.80)	8.378(8.31)	5.59(5.45)
TABS	C ₇₀ H ₅₄ N ₄ MoO ₆ S ₂	1207.29	50	145	69.64(69.50)	4.50(4.45)	4.644(4.55)	7.947(7.60)	5.315(5.22)
TABS	C ₆₈ H ₄₉ N ₄ MoO ₆ S ₃ F ₃	1267.29	50	128	64.44(64.38)	3.89(3.80)	4.424(4.35)	7.75(7.68)	7.59(7.51)
TABS	C ₇₅ H ₅₆ N ₄ MoO ₆ S ₂	1269.36	45	130	70.96(70.90)	4.44(4.20)	4.41(4.32)	7.55(7.49)	5.05(4.85)

Stoichiometric chunk of metal along with ligand are in a 1:1 ascertained by elemental analysis (Table 1)

Antibacterial activity assay

The employed method was agar-well diffusion to track the biological efficacy in the antibacterial domain of several bacterial strains, ranging from *S. aureus*, *B. subtilis*, *E. aerogene* to *S. typhi*, *in vitro*⁶¹⁻⁶³. Doxycycline started to have the same antibacterial action as other medicines. We used a metallic borer to drill holes in the agar media that were 6 mm in diameter. There were 3x10⁵ colony-forming units per milliliter (mL) in all bacterial suspensions. With the use of standardized suspensions, agar was enlarged. A solution of 300 g mL⁻¹ of the generated chemicals in 1% DMSO was used to analyze the sample. The evaluated samples were placed into each well. Remaining wells were occupied with 0.05 percent doxycycline inclusive of DMSO. The test for growth inhibition is conducted after the incubation at 35°C lasting for 35 hours.

RESULTS AND DISCUSSION

Infrared spectra

By refluxing a mixture of thenil, 1,1'-Binaphthyl-2,2'-diamine, Mo(acac)₂ in ethanol with a ratio of 1:2:1, dioxomolybdenum (VI) was created *in-situ*. The scheme illustrates the macrocyclic compounds that were synthesized. The MoO₂(VI) compounds and the separate ligands both showed many infrared bands.

These bands are highlighted in Table 2. The lowering of νC=N absorption frequencies has been used to demonstrate a macrocyclic molecule having N-atoms of the group -C=N bound to Mo⁶³⁻⁶⁷. In spectrum bands spanning from 1566 to 1670 cm⁻¹, individual ligands exhibit a C=N signal at 1670 cm⁻¹⁶⁴⁻⁶⁶. The Mo-N vibration, which has nothing to do with free ligands, may be responsible for it⁶⁸. The existence of two C=N signals at about 1710 cm⁻¹ causes thenil to establish a strong bond with diamines^{66,67}. The target is 3320 cm⁻¹ for an N-H_{asym} broad band and 3130 cm⁻¹ for a N-H_{sym} wide band. Since the NH group is not present, TABSBMo and TABSBMo s are identical. This suggests that the bonding process does not involve the NH group⁷¹. The d-orbital is most frequently utilized for bonding among dioxomolybdenum (VI) compounds, generating preferentially cis-dioxo groups. Due to C_{2v} symmetry, cis-[MoO₂]²⁺ exhibits both symmetric and asymmetric stretching at 890-990 and 962-972 cm⁻¹ in dioxomolybdenum(VI) compounds⁷². The vasymand νsym(O=Mo=O) dioxomolybdenum frequencies are linked to twin IR bands at the concerned wavelengths, correspondingly⁷²⁻⁷⁹. νsym dioxomolybdenum has higher vibrations than νasym(O=Mo=O)^{80,81}. The acetylacetonate bands close to 1550-1565 of C=O and 1455-1480 cm⁻¹ C=C vibrations⁸². 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 -NH₂group^{82,83}.

Table 2: IR spectral bands of the compounds

Compound	C=N	Mo-N	C=O(acac)	C=C(acac)	ν of		asym(N-H)	ym(N-H)
					asym(O=Mo=O)	sym(O=Mo=O)		
TABS	1670m	---	---	---	---	---	3320br	3130br
TABS _{Mo}	1566s	493m	1492m	1397s	915s	972s	---	3122br
TABS _{Moβs} ¹	1652m	550s	1552s	1465m	895m	965s	---	---
TABS _{Moβs} ²	1630s	558s	1555s	1470s	910m	970m	---	---
TABS _{Moβs} ³	1650s	565m	1550m	1455m	990m	965m	---	---
TABS _{Moβs} ⁴	1655m	540s	1560s	1472s	895s	962s	---	---

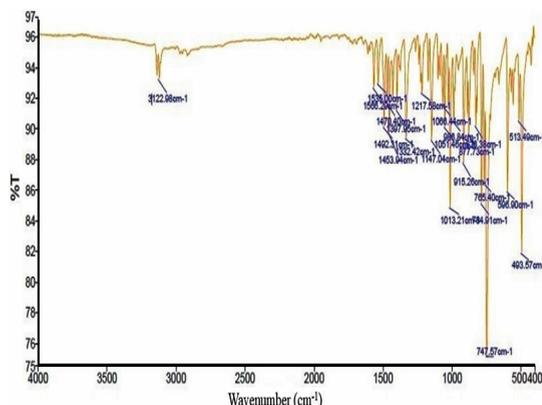


Fig. 3. IR spectrum of TABS_{Mo}

¹H NMR spectra

Table 3 represents all NMR signals. Holistic

NMR signals are accustomed to paragraphs. All ¹H NMR of MoO₂(VI) compounds in relation to the ligand were examined using DMSO-D₆. The cyclization via 1,3-diketones may be indicated by the presence of NH₂ at δ (5.49) in TABS, which is also present in TABS_{Mo} at δ (7.20) but absent in the rest compounds⁸⁷⁻⁸⁹. The ten protons in molybdenum compounds, including ligand, are multiplets in the δ (8.95-7.20) range. The protons of aromatic rings showed peaks at δ 8.30. Two varieties of azomethine are available for use in the macrocyclic compound's production, and they seem to cause these chemical changes. The bond to CH₃ of the diketone group may be the cause of the peak at δ 0.87.

Table 3: ¹H NMR data (in δ ppm)

Compound	HC-Binaphthyl	HC-thiophene	N-H	HC-CH ₃	HC-CH ₂	HC- thienyl	HC-Ar
TABS	8.06, 2H; 8.20, 2H; 7.39, 2H; 7.52, 2H; 8.95, 2H; 8.80, 2H; 7.25, 2H; 7.20, 2H; 7.97, 2H; 7.89, 2H; 7.76, 2H	7.53, 2H; 7.98, 2H; 7.01, 2H	5.49, 4H	---	---	---	---
TABS _{Mo}	8.06, 2H; 8.20, 2H; 7.39, 2H; 7.52, 2H; 8.95, 4H; 7.52, 2H; 7.39, 2H; 8.20, 2H; 8.06, 2H; 7.18, 2H	7.53, 2H; 7.69, 2H; 7.01, 2H	7.20, 4H	---	---	---	---
TABS _{Moβs} ¹	8.06, 2H; 8.20, 2H; 7.39, 2H; 7.52, 2H; 8.95, 4H; 7.52, 2H; 7.39, 2H; 8.20, 2H; 8.06, 2H; 7.69, 2H	7.53, 2H; 7.69, 2H; 7.01, 2H	---	0.87, 6H	1.05, 2H	---	---
TABS _{Moβs} ²	8.06, 2H; 8.20, 2H; 7.39, 2H; 7.52, 2H; 8.95, 4H; 7.52, 2H; 7.39, 2H; 8.20, 2H; 8.06, 2H; 7.69, 2H	7.53, 2H; 7.69, 2H; 7.01, 2H	---	0.87, 3H	1.05, 2H	---	7.30, 2H; 7.42, 2H; 7.45, 1H
TABS _{Moβs} ³	8.06, 2H; 8.20, 2H; 7.39, 2H; 7.52, 2H; 8.95, 4H; 7.52, 2H; 7.39, 2H; 8.20, 2H; 8.06, 2H; 7.69, 2H	7.53, 2H; 7.69, 2H; 7.01, 2H	---	---	1.05, 2H	7.15, 1H; 7.01, 1H; 7.53, 1H	---
TABS _{Moβs} ⁴	8.06, 2H; 8.20, 2H; 7.39, 2H; 7.52, 2H; 8.95, 4H; 7.52, 2H; 7.39, 2H; 8.20, 2H; 8.06, 2H; 7.69, 2H	7.53, 2H; 7.69, 2H; 7.01, 2H	---	---	1.05, 2H	---	7.30, 4H; 7.42, 4H; 7.45, 2H

UV-Visible spectra

The strong energy scheme proposed by the TABS and molybdenum(VI) compounds is in agreement with these spectra, which were obtained in ethanol^{84,85}. The spectra of molybdenum(VI) compounds and TABS 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) 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→π*/π→π* may be responsible for the strong absorption bands seen at 285 nm and 305 nm in those compounds' UV-Vis spectra.

$N(\pi) \rightarrow d(\text{Mo})$ appears to be the cause of the band between 370 and 385 nm. Electronic transition between LUMO of the Mo-atom and HOMO of the ligand is most facilitated by transition ${}^2B_2 \rightarrow {}^2A_1$ ($dxy \rightarrow dx^2-y^2$), which may be covered by other transitions. Using Ballhausen-Gray diagrams, the energy level of such compounds has been provided. The electronic spectra of each molecule is distorted by an octahedral pattern⁹⁰.

Magnetic and molar conductance measurements

It is not required to mention the diamagnetic nature of dioxomolybdenum (VI) compounds in the d^0 configuration. Since d-orbitals lack electrons in such compounds, no d-d transitions are found. 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 molar conductance of such substances fluctuates between 92 and $115 \Omega^{-1} \text{cm}^2 \text{mol}^{-1}$. Above compounds of type TABSBMo and TABSBMo s, the molar conductance values indicate tentative forms, as the schemes.

Thermogravimetric analyses

According to the compound's thermogravimetric curve (TG Curve), the compound

broke down in a single step. The loss of the TABSB and acetylacetonate (estimated weight of 83.1%) is the cause of the observed 83% weight loss at 279°C. MoO_3 makes up 16.3% of the residual at 281°C. MoO_2 is responsible for the 14.48% weight remaining at 324°C.

Antibacterial activity

The dioxomolybdenum (VI) compounds have antibacterial action, as shown in Table 4. $\text{MoO}_2(\text{VI})$ complexes showed bactericidal activity against a range of bacteria. We have taken a variety of bacterial like *S. aureus*, *B. subtilis*, *E. aerogenes*, and *S. typhi*. The enhanced antibacterial activities of compounds can be explained by the chelation hypothesis^{82,83}. A doxycycline cure serves as the source of information. The activity of many of the compounds in response to *S. aureus* and *S. typhi* was found to be low or moderate.

Employing Eq. 1 at the outset, an estimation of the inhibition effect (%) was executed depending on the +ve control

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

Table 4: Antibacterial activities of compounds*

Compound	<i>S. aureus</i>	<i>E. aerogenes</i>	<i>S. typhi</i>	<i>B. subtilis</i>	Doxycycline
TABSBMo	16	19	19	22	25
TABSBMo β s ¹	14	20	18	20	23
TABSBMo β s ²	15	20	18	20	24
TABSBMo β s ³	13	19	17	--	24
TABSBMo β s ⁴	17	18	--	21	23

*Inhibition zone activity (mm): 23-26 = strong; 15-18 = moderate; 19-22 = good; doxycycline as a reference drug

InChI

InChI=1S/C50H34N4S2.Mo.2O/c51-40-23-19-31-10-1-5-14-36(31)45(40)47-38-16-7-3-12-33(38)21-25-42(47)53-49(35-27-29-55-30-35)50(44-18-9-28-56-44)54-43-26-22-34-13-4-8-17-39(34)48(43)46-37-15-6-2-11-32(37)20-24-41(46)52;;;/h1-30H,51-52H2;;;/b53-49+,54-50+;;;

$C=C7)=C(C(C8=CSC=C8)=[N+])1C9=C(C(C=CC=C\%10)=C\%10C=C9)C\%11=C([NH2+])3C=CC\%12=C\%11C=CC=C\%12)C\%13=CC=CS\%13)=O$

LogP

0

TABSBMoInChI key

CGKRJICCAFQBHX-VQOQNKZSA-N

TABSBMoSMILES

$O=[\text{Mo}-4]123([N+](C(C=C4)=C(C5=C([NH2+])2)C=CC6=C5C=CC=C6)C7=C4C=C$

CONCLUSION

Since their crystals have not been separated, we are unable to provide crystal structures for the corresponding compounds. It is feasible to represent all of the produced compounds as TABSBMo or

TABSBMo β s using the elemental and spectral research mentioned above. Antibacterial activity tests have been conducted on these substances to evaluate their biological activity. The present study demonstrates dioxin derivatives produced from the TABSB of Mo(VI). 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 TABSB. The metal ion and ligand ratio is 1:1, according to analytical data. The produced compounds have been proposed to have deformed octahedral and six-coordinated geometries and a mononuclear.

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Conflict of Interest

Authors declare no competent financial interest.

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