



Dioxomolybdenum (VI) Compounds of Macrocyclic Schiff base Ligands: Preparation, Characterization and Antibacterial activity

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ABSTRACT

Using di-2-furanylethanedione and 5-bromo-3-methylbenzene-1,2-diamine we prepared a monomeric [MoO₂(SL)] with a Schiff base, as well as 4 different compounds using the formulation [MoO₂(MSL)]. We investigate how [MoO₂(SL)] reacts with 1,3-diketones. Several characterizations are discussed in this article, including molar conductance measurement, elemental analysis, UV-Vis, IR, NMR, and thermal measurements. Molybdenum has a six-coordination number. All five MoO₂(VI) compounds have distorted octahedral arrangements. Molybdenum octahedra have four N-atoms and two oxidized O-atoms. Against *S. aureus* and *S. typhi*, all synthesized compounds showed moderate activity. The chelation hypothesis is used to define the progression of the antibacterial task.

Keywords: Dioxomolybdenum(VI), 5-bromo-3-methylbenzene-1,2-diamine, Schiff base, Di-2-furanylethanedione, β-diketones, Macrocyclic compounds, Antibacterial activity.

INTRODUCTION

At least nine atoms are required for a macrocyclic compound, including all heteroatoms. This type of compound contains at least three donor atoms. Over the past half century, macrocyclic compounds have undergone considerable development¹⁻⁵. In coordination chemistry, N-atom-containing ligands and their compounds play an

important role. There have been various research papers published that explore metal compounds from physicochemical and biochemical perspectives and drive extensive applications⁶⁻¹¹. A transitional Schiff base formulation with vitamin B6 catalyzes transamination reactions using metal ions^{15,16}. In addition to their antifungal and antiviral properties, Schiff base compounds are also anti-inflammatory and antitumor¹²⁻²⁰.



Metalloproteins and enzymes contain positions of metal that can be modelled by Schiff base compounds in the bioinorganic field²¹. In comparison to the isolated ligand, the anticancer activity of various Schiff bases is greater for that particular metal compound²²⁻²⁹. Additionally, they remain fascinated by chemistry reactions and consumer electronics memory storage gadgets.

Extreme density ligands in transition metal compounds have different configurations and coordination numbers^{30,31}. The multiple oxidation states of molybdenum can allow it to be adaptable in this aspect. A coordination number may also range from 4 to 8³². Schiff base edifices can be produced for homogeneous and heterogeneous reactions by linking molybdenum compounds with ligands containing hetero atoms (N,O and S)³³⁻³⁹. There are a number of biological applications that can be achieved by MoO₂(VI) compounds because they have multidentate ligands. [MoO₄]²⁻ can readily be acquired in aqueous solution as Mo(VI). Solution concentration and pH can determine the concentration of the [MoO₄]²⁻ ion. A template for oxygen transfer has been shown to exist in the form of the [MoO₄]²⁻ ion. It has been shown that their oxygen transfer properties have a negative effect on the molybdenum oxotransferase mechanism⁴⁰⁻⁴³. It is known that several redox enzymes are fully oxidized. A cis-dioxomolybdenum moiety is associated with their active sites in such cases⁴⁴⁻⁴⁶.

Molybdenum is the only transition metal considered as a biometal that is essential for pathogenic microorganisms found in human, animal and vegetation pathogens^{47,48}. The biological and catalytic properties of Mo(VI) coordinate chemistry make it an active area of scientific research⁴⁹⁻⁵². A molybdenum molecule sets up the physiological functions of oxomolybdoenzymes⁵³⁻⁵⁵. It might be useful as a chelating agent since di-2-furanylethanedione is versatile. There could be Schiff base condensation between diamines and di-2-furanylethanedione due to its reactive carbonyl groups. In order to synthesize macrocyclic ligands, di-2-furanylenedione plays an essential role.

Di-2-furanylethanedione has denticity

ligands, which can be synthesized from reaction of diamine with di-2-furanylethanedione under certain conditions. Through metal template impact, the synthesized compound undergoes cyclization with 1,3-diketones. It is possible to prepare and characterize them, and TGA, nmr, ir, UV-Vis spectroscopy, molar conductivity measurements, and elemental analysis have all supported their provisional structures.

EXPERIMENTAL

Materials

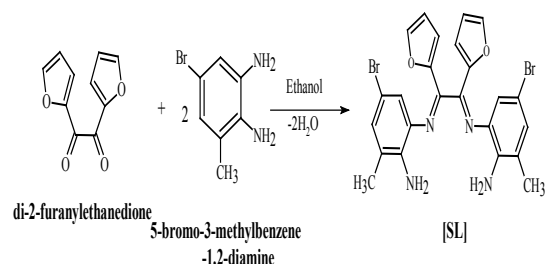
Chemists obtained from business resources prepared Schiff bases (ligands) and compounds using reagent grade chemicals. In addition to molybdenyl acetylacetonate, 5-bromo-3-methylbenzene-1,2-diamine, and di-2-furanylethanedione, 1,3-diketones were obtained from Aldrich without similar refinement.

Physical Measurements and Analytical Methods

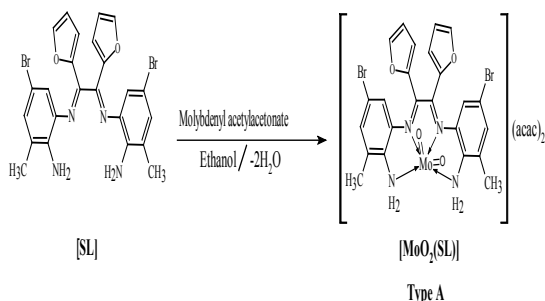
In Arunachal Pradesh, India, CHN analysers were used at important CRFs, including NERIST, Nirjuli, Itanagar, to examine C,H and N in the compounds. Nitrogen is assessed using Kjeldahl's method for synthesized compounds. Using a gravimetric technique, molybdenum was measured after decay of the compound⁵⁶. As barium sulfate was developed as a method for estimating sulfur, sulfur estimation progressed⁵⁷. In addition to sulfuric acid baths, general methods were used for determining uncorrected melting factors. A Labinda-UV 3000+ UV/Vis spectrophotometer at UPTTI Kanpur, U.P., India was used to compute the electronic absorption spectra of the compounds utilizing ethanol. At Indian Institute of Technology Kanpur, we recorded the infrared spectra of MoO₂(VI) compounds in KBr using Perkin-Elmer Spectrum models 10.03.06 and 18.03.06 spectrophotometers. On the JMM ECS-400 (JEOL) spectrometer, proton (¹H NMR) spectra of MoO₂(VI) compounds were obtained. An analysis of the temperature distribution of the [MoO₂(SL)] compound was carried out using the TG/DTA-SDT Q600 V 20.9 Built 20, USA thermal analyzer under a nitrogen atmosphere at temperatures between 50-900°C at a 10°C min⁻¹ heating rate.

Preparation of molybdenum(VI) compounds containing ligands that result from condensation of di-2-furanylethanedione and 5-bromo-3-methylbenzene-1,2-diamine with 1,3-diketones.

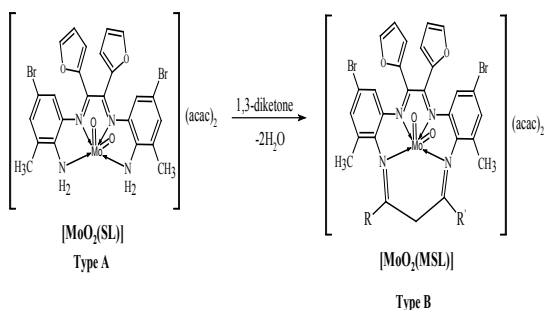
Schemes 1, 2 and 3 show how to synthesize $[\text{MoO}_2(\text{MSL})]$. Drop-wise addition of molybdenyl acetylacetonate (1.6307 g, 5 mmol) in ethanol (50 mL) was followed by the addition of di-2-furanylethanedione (0.9506 g, 5 mmol) in 50 mL ethanol and 5-bromo-3-methylbenzene-1,2-diamine (2.0106 g, 10 mmol) in 50 mL ethanol. The solution turns brown after 3 h of slight reflux in the reaction mixture. After filtering, ethanol was used to wash the solid product, and silica gel was used to isolate the solid under vacuum. In order to check the purity of the compound, the TLC method was used. As a result, the yield increased to 48% (type A). 3D structures of the Schiff base ligand (SL) and parent compound $[\text{MoO}_2(\text{SL})]$ are given in Figure 1 and 2.



Scheme 1. Synthesis of Schiff base ligand (SL)



Scheme 2. Synthesis of $[\text{MoO}_2(\text{SL})]$



Scheme 3. Synthesis of $[\text{MoO}_2(\text{MSL})]$

Where, SL = di-2-furanylethanedione+ 5-bromo-3-methylbenzene-1,2-diamine; MSL = in the presence of dioxmolybdenum(VI) cation, macrocyclic ligands are synthesized by condensation of SL with 1,3-diketones.

MSL¹: 2R = CH₃, R' = CH₃; MSL²: R = C₆H₅, R' = CH₃; MSL₃: R = C₄H₉S, R' = CF₃; MSL⁴: R = C₆H₅, R' = C₆H₅

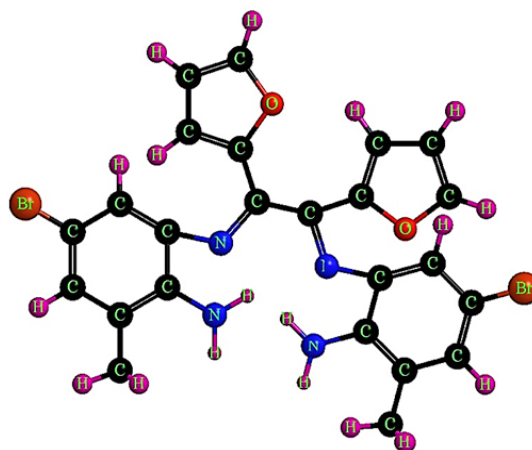


Fig. 1. 3D structure of Schiff base ligand (SL)

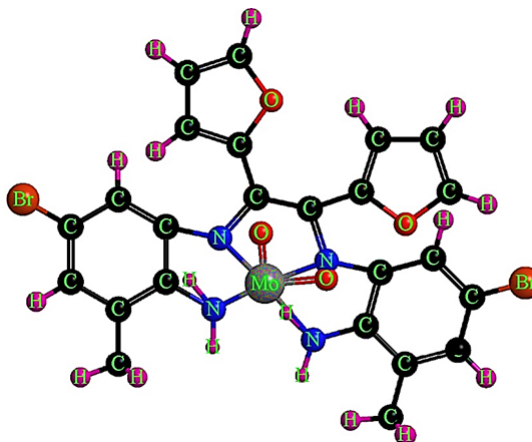


Fig. 2. 3D structure of parent compound $[\text{MoO}_2(\text{SL})]$

Upon addition of 1,3-diketones to ethyl alcohol suspension of Kind A, a uniform reaction was observed after three hours. Here we used 1,3-diketones like 4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedione, 1,3-diphenyl-1,3-propanedione, 2,4-pentanedione or 1-phenyl-1,3-butanedione (1:1) to produce yellow solid macrocyclic stable products (type B). A TLC test is performed to verify the purity of macrocyclic compounds.

Where,

Table 1: Schiff base ligands and compounds: physical and analytical data

Compound	Formula	F.W.	Yield (%)	m.p. (°C)	% of Element, Calcd./(found)				
					C	H	N	Mo	S
SL	$C_{24}H_{20}Br_2N_4O_2$	556.26	65	120	51.82 (51.74)	3.62 (3.60)	10.07 (10.00)	--	--
[MoO ₂ (SL)]	$C_{34}H_{34}Br_2N_4MoO_8$	882.41	55	118	46.27 (46.20)	3.88 (3.78)	6.34 (6.38)	10.87 (10.80)	--
[MoO ₂ (MSL ₁)]	$C_{39}H_{38}Br_2N_4MoO_8$	946.52	50	130	49.49 (49.40)	4.05 (4.00)	5.92 (6.86)	10.14 (10.9)	--
[MoO ₂ (MSL ₂)]	$C_{44}H_{40}Br_2N_4MoO_8$	1008.59	45	155	52.40 (52.33)	4.00 (3.96)	5.56 (5.32)	9.51 (9.46)	--
[MoO ₂ (MSL ₃)]	$C_{42}H_{38}Br_2N_4MoO_8F_3S$	1071.59	45	125	47.05 (46.90)	3.57 (3.50)	5.22 (5.15)	8.95 (8.89)	2.99 (2.95)
[MoO ₂ (MSL ₄)]	$C_{49}H_{42}Br_2N_4MoO_8$	1070.66	50	132	54.97 (54.94)	3.95 (3.91)	5.23 (5.20)	8.96 (8.90)	--

SL= Di-2-furanylethanedione has been transformed into a Schiff base by condensation with 5-bromo-3-methylbenzene-1,2-diamine; Ligand MSL¹ synthesized via condensation of SL with 2,4-pentanedione to form a macrocyclic Schiff base; Ligand MSL² synthesized via condensation of SL with 1-phenyl-1,3-butanedione to form a macrocyclic Schiff base; Ligand MSL³ synthesized via condensation of SL with 4,4,4-trifluoro-1- (2-thienyl)-1,3-butanedione to form a macrocyclic Schiff base; Ligand MSL⁴ synthesized via condensation of SL with 1,3-diphenyl-1,3-propanedione to form a macrocyclic Schiff base.

According to elemental analysis (Table 1), metal and ligand are in a 1:1 stoichiometry.

Antibacterial activity assay

Several bacterial strains were tested *in vitro* for their antibacterial activity, including *S. aureus*, *B. subtilis*, *E. aerogene* and *S. typhi* utilizing an agar-well diffusion process⁵⁸⁻⁶⁰. Antibacterial activity of Doxycycline became the same as that of other antibiotics. Agar media were drilled with 6 mm-diameter holes using a metallic borer. All bacterial suspensions were adjusted to a density of 3×10^5 colony-forming units per mL. Agar was expanded with standardized suspensions. In the examined sample, 300 g mL⁻¹ of the produced compounds were dissolved in 1% DMSO. Each well was filled with the examined samples. DMSO and Doxycycline (0.05%) were packed in the rest of the wells. Upon incubation at 355°C for 30 h growth inhibition is tested.

RESULTS AND DISCUSSION

Infrared spectra

Through an *in-situ* procedure, dioxomolybdeum(VI) was produced by refluxing a mixture of di-2-furanylethanedione, 5-bromo-3-methylbenzene-1,2-diamine, and molybdenyl acetylacetonate in aqueous ethanol in a 1:2:1 ratio, which resulted in macrocyclic compounds as shown in the scheme. Several infrared bands were observed for the isolated ligands and the MoO₂(VI) compounds. Table 2 highlights these bands. A macrocyclic compound with N-atoms of the group azomethine bonded to molybdenum has been demonstrated by lowering C=N absorption frequencies⁶¹⁻⁶⁴. In isolated ligands, >C=N absorption appears at 1665 cm⁻¹ in spectrum bands between 1610-1650 cm⁻¹⁶¹⁻⁶³. It could be assigned to the Mo-N vibration around 490-570 cm⁻¹, which is not related to free ligands⁶⁵. Di-2-furanylethanedione forms a strong bond with diamines due to the presence of two >C=N bands around 1710 cm⁻¹^{66,67}. Dioxomolybdeum(VI) compounds and their isolated ligands exhibit compound infrared spectra due to various ring vibrations and C-H vibrations. An asymmetric (N-H) and symmetric (N-H) wide band is targeted at 3430 and 3060 cm⁻¹, respectively. There is no difference between [MoO₂(SL)] and [MoO₂(MSL)] because the NH group is absent. This implies the NH group is not involved in the bonding process⁶⁸. Among dioxomolybdenum(VI) compounds, d-orbital is used most often for bonding, forming preferentially

cis-dioxo groups. In dioxomolybdenum(VI) compounds, $\text{cis-[MoO}_2\text{]}^{2+}$ demonstrates asymmetric and symmetric stretching vibrations at $896\text{--}910\text{ cm}^{-1}$ and $962\text{--}970\text{ cm}^{-1}$ as a result of C_{2v} symmetry⁶⁹. There are two infrared spectral bands at these wavelengths that are associated with $\nu_{\text{asym(O=Mo=O)}}$ and $\nu_{\text{sym(O=Mo=O)}}$ vibrations respectively⁶⁹⁻⁷⁶. In comparison to $\nu_{\text{sym(O=Mo=O)}}$, $\nu_{\text{asym(O=Mo=O)}}$ vibrations are lower^{77,78}. On the outer coordination sphere,

the acetylacetonate group can be observed in the bands around $1555\text{--}1570\text{ cm}^{-1}$ and $1465\text{--}1482\text{ cm}^{-1}$, which correspond to the C=O and C=C vibrations, respectively⁷⁹. The macrocyclic compounds exhibit an equal pattern of infrared spectral bands. A method of cyclizing 1,3-diketones by containing amino groups on the carbonyl group, the asymmetrically and symmetrically stretching vibrations of terminal amino groups disappear^{79,80}.

Table 2: Schiff base ligand and dioxomolybdenum compounds IR spectral bands (ν/cm^{-1})

Compound	C=N	Mo-N	C=O _(acac)	C=C _(acac)	ν of			
					$\nu_{\text{asym(O=Mo=O)}}$	$\nu_{\text{sym(O=Mo=O)}}$	$\nu_{\text{asym(N-H)}}$	$\nu_{\text{sym(N-H)}}$
SL	1660m	---	---	---	---	---	3325br	3133br
[MoO ₂ (SL)]	1610s	490m	1570m	1465s	901s	965s	3430br	3060br
[MoO ₂ (MSL ₁)]	1642m	555s	1565s	1480m	905m	965s		
[MoO ₂ (MSL ₂)]	1650s	562s	1560s	1472s	910m	970m		
[MoO ₂ (MSL ₃)]	1630s	570m	1555m	1470m	904m	972m		
[MoO ₂ (MSL ₄)]	1645m	530s	1563s	1470s	896s	965s		

In the range of $4000\text{--}400\text{ cm}^{-1}$, all spectra were recorded with KBr

¹H NMR spectra

All NMR signals are given in Table 3. All MoO₂(VI) compounds as well as the ligand were analyzed using ¹H NMR in DMSO-*d*₆. It is possible that SL contains signal because of NH₂ at δ (5.32), which is also found in [MoO₂(SL)], at δ (7.20) but not in other macrocyclic compounds, which suggests the presence 1,3-diketones that are present in

cyclization⁸⁴⁻⁸⁶. For isolated ligands and molybdenum compounds, the ten protons are multiplets within the range δ (7.84-6.53). About δ 7.25 peaks appeared for the protons of aromatic rings. In the preparation of the macrocyclic compound, two types of azomethine are offered which appear to result in these chemical shifts. It is possible that peak at δ 2.33 is be due to CH₃ attached to an aryl group.

Table 3: Dioxomolybdenum compounds and ligand ¹H NMR Spectral Data (in δ ppm)

Compound	HC-Ar	N-H	H ₃ C-Ar	=C-CH ₃	-CH ₂	HC-furan	HC- thienyl
SL	7.12 2H7.17 2H	5.32 4H		2.12 6H	--	6.54 4H7.84 2H	--
[MoO ₂ (SL)]	7.32 2H7.40 2H	7.20 4H		2.33 6H	--	6.54 4H7.84 2H	--
[MoO ₂ (MSL ₁)]	7.32 2H7.40 2H	--	2.33 6H	0.87 6H	1.05 2H	6.54 4H7.84 2H	--
[MoO ₂ (MSL ₂)]	7.32 4H7.40 5H	--	2.33 6H	0.87 3H	1.05 2H	6.54 4H7.84 2H	--
[MoO ₂ (MSL ₃)]	7.32 2H7.40 2H	--	2.33 6H	0.87 3H	1.05 2H	6.54 4H7.84 2H	7.01 1H7.15 1H7.53 1H
[MoO ₂ (MSL ₄)]	7.32 6H7.40 8H	--	2.33 6H	--	1.05 2H	6.54 4H7.84 2H	--

UV-Visible spectra

These spectra were measured in ethanol, and they are consistent with the strength energy level scheme suggested by the tetradentate tetraaza ligand and dioxomolybdenum(VI) compounds⁸¹⁻⁸³. Tetradentate ligands and dioxomolybdenum(VI) compounds exhibit similar spectra. With the Mo(VI) ion having no d-electron, it is no longer expected to show pure d-d absorption bands. All molybdenum compounds can also be explained as charge transfer transitions between their nitrogen and molybdenum d-orbitals [N(π) \rightarrow d(Mo)]. A dioxomolybdenum(VI) compound containing nitrogen donor atoms exhibits

homogeneous UV-Vis spectra. Strong absorption bands during the UV-Vis spectrum of those compounds is identified at 292 nm and at 311 nm, which may be attributed to intraligand transitions and $n\rightarrow\pi^*/\pi\rightarrow\pi^*$ transitions. The band between 380 nm and 395 nm appears to be caused by N(π) \rightarrow d(Mo). Transition ${}^2B_2\rightarrow{}^2A_1$ ($d_{xy}\rightarrow d_{x^2-y^2}$) may be covered within other bands and is ideal to facilitate charge transfer between the molybdenum LUMO and the ligand HOMO. Energy level schemes for these compounds have been provided by Ballhausen-Gray diagrams. Each compound's electronic spectrum has an octahedral distortion⁸⁷.

Magnetic and molar conductance measurements

As for d^0 configuration, it is not necessary to mention that dioxomolybdenum(VI) compounds are diamagnetic. Due to the absence of electrons in d-orbitals, no d-d transitions are determined for those compounds. The molar conductivities (Λ_m) of all molybdenum compounds in DMF at the ca. 10^{-3} M endorse 1:1 sort electrolytes. Those compounds have molar conductance ranging from 90 to $110 \Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$. As predicted by the schemes, the molar conductance values on top of the compounds of dioxomolybdenum(VI) type (A) and macrocyclic type (B) suggest tentative shapes.

Thermogravimetric analyses

Thermogravimetric curve (TG Curve) of the compound presented in Fig. 3 reveals that the compound has decomposed in one step. At a temperature of 279 about 83% weight loss has been observed which is due to loss of Schiff ligand and acetylacetonate moiety (calculated weight 83.1%). At 281, the residue left is 16.3% which corresponds to MoO_3 . Weight percent left at 324 is 14.48 and is attributed to MoO_2 .

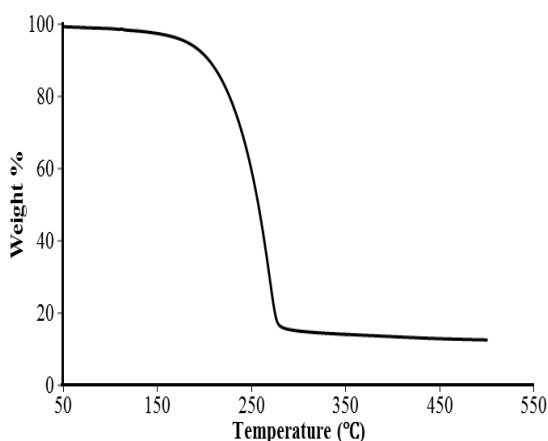


Fig. 3. TG thermograph of $[\text{MoO}_2(\text{SL})]$

Antibacterial activity

As can be seen in Table 4, the dioxomolybdenum(VI) compounds exhibit

antimicrobial activity. It was tried against a variety of bacteria, including *S. aureus*, *B. subtilis*, *E. aerogenes*, and *S. typhi*, using the synthesized dioxomolybdenum(VI) compounds. Chelation hypothesis can be used to explain the improvement of dioxomolybdenum(VI) compounds' antibacterial properties^{79,80}. A doxycycline remedy is used as the reference material. Many of the compounds were found to have low or modest activity against *S. aureus* and *S. typhi*.

Using Eq 1 as a starting point, we calculate the percentage of inhibition effect based on the positive control.

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

^aIn vitro diffusion method using cups and wells conc. $300 \mu\text{g mL}^{-1}$ in solvent DMSO; Inhibition zone (mm): 23-26 = strong activity; 15-18 = moderate activity; 19-22 = good activity; an antibiotic drug of reference is doxycycline.

InChI

InChI=1S/C₂₄H₂₀Br₂N₄O₂/c1-13-9-15(25)11-17(21(13)27)29-23(19-5-3-7-31-19)24(20-6-4-8-32-20)30-18-12-16(26)10-14(2)22(18)28/h3-12H,27-28H2,1-2H3/b29-23+,30-24+

InChI key of compound

VWNWPFYNPOEBGW-HCTXVGCHSA-N

SMILES Notation

Brc4cc(\N=C(\C(=N\c1cc(Br)cc(C)c1N)\c2ccco2)c3ccco3)c(N)c(C)c4

LogP

5.50+/- 0.67

Table 4: Antibacterial activitiesa

Compound	<i>S. aureus</i>	<i>E. aerogenes</i>	<i>S. typhi</i>	<i>B. subtilis</i>	Doxycycline
$[\text{MoO}_2(\text{SL})]$	17	20	17	21	26
$[\text{MoO}_2(\text{MSL}_1)]$	13	20	18	20	24
$[\text{MoO}_2(\text{MSL}_2)]$	13	19	17	22	25
$[\text{MoO}_2(\text{MSL}_3)]$	14	19	19	--	24
$[\text{MoO}_2(\text{MSL}_4)]$	16	17	--	22	23

CONCLUSION

These compounds haven't been crystallized yet, so their crystal structures are unavailable. The [MoO₂(SL)] and [MoO₂(MSL)] compounds can be represented using the above elemental and spectral studies. The antibacterial activity of these compounds has been tested. We demonstrate the formation of dioxin derivatives from dioxomolybdenum(VI) Schiff bases. The Schiff base condensation was confirmed using a flexible chelator with two carbonyl groups, di-2-furanylethanedione. As a result of their reaction with 1,3-diketones, macrocyclic products are formed. Distorted octahedron surrounding Mo. Di-2-furanyledione and diamines are used to condense Schiff bases in ethanol medium. Tetradentate bonds are formed between azomethine nitrogen atoms and metal ions in Schiff bases. One metal ion is present in each ligand molecule. Compounds prepared have been shown to have six coordinated

distorted octahedral shapes and a mononuclear six coordination.

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Conflict of Interest

Financial interests of the authors are not in conflict.

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