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# Alkali Metal Complexes of an Octamethyl Isomeric Macrocycle: Synthesis, Characterization and Antimicrobial Studies

Avijit Chakraborty[1], Saswata Rabi[1,2], Snehashis Roy[1], Debashis Palit[1],
Zinnat Ara Begum[3,4], Ismail M.M. Rahman[3,*] and Tapashi Ghosh Roy[1,*]

[1]Department of Chemistry, Faculty of Science, University of Chittagong, Chattogram 4331, Bangladesh
[2]Department of Chemistry, Faculty of Engineering & Technology, Chittagong University of Engineering and Technology, Chattogram 4349, Bangladesh
[3]Institute of Environmental Radioactivity, Fukushima University, 1 Kanayagawa, Fukushima City, Fukushima 960-1296, Japan
[4]Department of Civil Engineering, Southern University Bangladesh, Arefin Nagar, Bayezid Bostami, Chattogram 4210, Bangladesh

*Corresponding authors: E-mail: immrahman@ipc.fukushima-u.ac.jp; tapashir57@cu.ac.bd

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Fourteen membered octamethyl tetraazamacrocyclic ligand salt, $Me_8[14]diene\cdot 2HClO_4$ and three isomeric ligands ($L_A$, $L_B$ and $L_C$) of its saturated analog have been synthesized as per literature. Interaction between the isomeric ligand $L_B$ and KX (X = SCN, $NO_3$, Cl, Br and I) afforded white-coloured five coordinated square pyramidal potassium complexes [$KL_BX$]. Moreover, the reaction between RbCl and $L_B$ produced five coordinated [$RbL_BCl$] having square pyramidal geometry. Then, the axial substitution reaction on [$RbL_BCl$] furnished a substituted rubidium complex with the same geometry of the mother complex. All the alkali metal complexes have been characterized by using modern analytical techniques. Furthermore, antibacterial and antifungal activities of ligand $L_B$ and its alkali metal (K and Rb) complexes have been investigated against different bacteria and fungi.

**Keywords: Alkali metals, Tetraazamacrocyclic complexes, Isomeric ligands, Biological activities.**

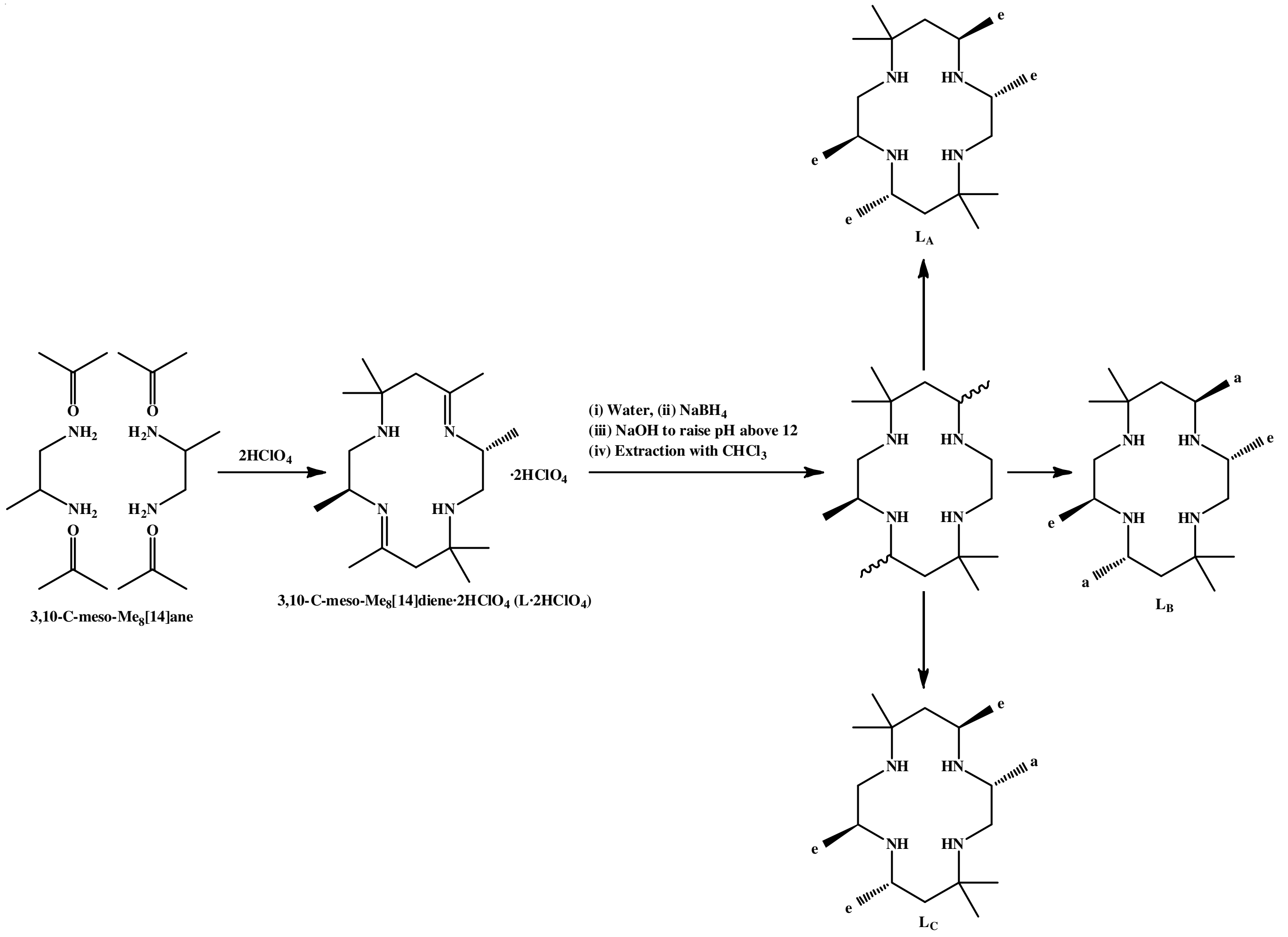
## INTRODUCTION

The metal complexes of macrocyclic ligands play an important role in many areas of science, and as a result, they have risen to prominence in the scientific community [1-3]. Macrocyclic compounds are showing their capability as antimicrobial [4], antitumor [5] and anticancer [6] agents. Moreover, these compounds are also used in magnetic resonance imaging [7] and radioimmunotherapy [8]. Efforts have been made to synthesize new macro-cyclic complexes of varying alkali metals and reports available for complexes with Cs(I), Li(I), Na(I), K(I), Rb(I) as metal templates [9-13]. The coordination chemistry of some alkali metal compounds has been established by X-ray crystallography [9,13]. We recently reported X-crystallographic data where Na(I) got coordinated [13]. In the current work, we attempted to prepare some new macrocyclic compounds of K(I) and Rb(I) with an octamethyl isomeric macrocycle, $L_B$ (an isomer of $Me_8[14]ane$), because different metal complexes of $L_B$ have been reported whereas alkali metal complexes of $L_B$ have not been reported by far. Isomeric ligand $L_B$ has been synthesized (**Scheme-I**) as per literature [14]. The current report includes the synthesis, characterization and antimicrobial potential data of the complexes of K(I) and Rb(I) with $L_B$.

## EXPERIMENTAL

Elemental analyses (C, H, N) were performed on a Leco CHNS-932 elemental analyzer (LECO Corporation, St. Joseph, MI). IR spectra were recorded on a Shimadzu IR 20 spectrophotometer (Shimadzu, Kyoto, Japan) as KBr disks. $^1$H NMR measurement was carried out on a Bruker AVANCE 400 spectrometer (Bruker AG, Karlsruhe, Germany). Conductance measurements were conducted on a conductivity bridge HI-8820 (Hanna Instruments, Padova, Italy).

The parent octamethyl substituted ligand salt, $L\cdot 2HClO_4$ and three isomers $L_A$, $L_B$ and $L_C$ of its reduced analog were synthesized (**Scheme-I**), following the method reported elsewhere [14].



**Scheme-I:** Preparation of isomeric ligand $L_B$

### Potassium complexes were prepared by the direct reaction of $L_B$ (an isomer of $Me_8$[14]ane) with K(I) salts

**[$KL_B$(NCS)]:** Potassium thiocyanate (0.194 g, 2.0 mmol) and isomeric ligand $L_B$ (0.312 g, 1.0 mmol) were dissolved separately in 20 mL of hot methanol, then mixed both the solutions and finally refluxed the reaction mixture for 11 h. The reaction mixture was filtered and the filtrate was allowed to stand for two days for slow evaporation. The white product of [$KL_B$(NCS)] was separated by filtration, followed by washing with methanol and diethyl ether. The dried product was stored in a vacuum desiccator over silica gel. Colour: white; yield: 54%. Anal. calcd. (found) % of $C_{19}H_{40}N_5KS$ (*m.w.*: 409.72): C, 55.69 (55.61); H, 9.04 (8.99); N, 17.09 (17.02). IR (KBr disc, cm$^{-1}$): ν(N–H), 3253s; ν(C–H), 2982s; ν($CH_3$), 1395s; ν(C–C), 1143vs; ν(CN), 2056 s, ν(CS), 831 s, δ(NCS), 470 m. Molar conductivity in DMSO: 30 ohm$^{-1}$ cm$^2$ mol$^{-1}$ and in DMF: 38 ohm$^{-1}$ cm$^2$ mol$^{-1}$. $^1$H NMR (400 MHz, DMSO, 25 ºC, TMS): For $CH_3$, δ (ppm) = 1.23 (s, 6H, e), 1.36 (s, 6H, a); Methyl on chiral carbon δ (ppm), 1.16 (d, 6H, e), 1.32 (d, 6H, a). For $CH_2$ & NH, = 1.70 (d, 4H), 1.87 (m), 2.26 (m), 2.75 (m), 3.24 (m). Magnetic moment $\mu_{eff}$ (B.M.): Diamagnetic.

**Synthesis of [$KL_B$($NO_3$)], [$KL_B$Cl], [$KL_B$Br] and [$KL_B$I]:** The complexes, [$KL_B$($NO_3$)], [$KL_B$Cl], [$KL_B$Br] and [$KL_B$I] were also synthesized as per the procedure described above using $KNO_3$, KCl, KBr and KI, respectively in a required ratio instead of KSCN.

**[$KL_B$($NO_3$)]:** Colour: white; yield: 48%. Anal. calcd. (found) % of $C_{19}H_{40}N_5KO_3$ (*m.w.*: 413.64): C, 52.26 (52.30); H, 9.74 (9.72); N, 16.93 (16.88). IR (KBr disc, cm$^{-1}$): ν(N–H), 3270s; ν(C–H), 2974m; ν($CH_3$), 1390m; ν(C–C), 1163s, ν($NO_3$), 1437w, 1321w. Molar conductivity in DMSO: 39 ohm$^{-1}$ cm$^2$ mol$^{-1}$. Magnetic moment $\mu_{eff}$ (B.M.): Diamagnetic.

**[$KL_B$Cl]:** Colour: white; yield: 63%. Anal. calcd. (found) % of $C_{18}H_{40}N_4KCl$ (*m.w.*: 387.09): C, 55.85 (55.81); H, 10.42 (10.39); N, 14.47 (14.40). IR (KBr disc, cm$^{-1}$): ν(N–H), 3258s; ν(C–H), 2962m; ν($CH_3$), 1383m; ν(C–C), 1142s. Molar conductivity in DMSO: 37 ohm$^{-1}$ cm$^2$ mol$^{-1}$. Magnetic moment $\mu_{eff}$ (B.M.): Diamagnetic.

**[$KL_B$Br]:** Yield: 61%. ($C_{18}H_{40}N_4KBr$), (431.54); Colour: white. Anal. calcd. (%): C, 50.09; H, 9.34; N, 12.98. Found: C, 50.02; H, 9.29; N, 12.90. IR (KBr disc, cm$^{-1}$): ν(N–H), 3259s; ν(C–H), 2962m; ν($CH_3$), 1383s; ν(C–C), 1142s. Molar conductivity (ohm$^{-1}$ cm$^2$ mol$^{-1}$) in DMSO, 22. Magnetic moment $\mu_{eff}$ (B.M.): Diamagnetic.

**[$KL_B$I]:** Colour: white; yield: 64%. Anal. calcd. (found) % of $C_{18}H_{40}N_4KI$ (*m.w.*: 478.54): C, 45.18 (45.12); H, 11.71 (11.64); N, 8.43 (8.39). IR (KBr disc, cm$^{-1}$): ν(N–H), 3280sh; ν(C–H), 2950w; ν($CH_3$), 1383m; ν(C–C), 1162s. Molar conductivity in DMSO: 32 ohm$^{-1}$ cm$^2$ mol$^{-1}$. Magnetic moment $\mu_{eff}$ (B.M.): Diamagnetic.

### Rb(I) complexes of $L_B$

**[RbL$_B$Cl]:** Rubidium(I) chloride (0.120 g, 1.0 mmol) and isomeric ligand $L_B$ (0.312 g, 1.0 mmol) were dissolved separately in 20 mL of hot methanol, then mixed both the solutions and finally refluxed the reaction mixture for 6 h. The reaction mixture was then filtered and the filtrate was allowed to stand for two days after concentrating to about 10 mL. A white product of [RbL$_B$Cl] was separated by filtration and washed with methanol and diethyl ether. Finally, the product was stored in a vacuum desiccator over silica gel.

**[RbL$_B$Cl]:** Colour: white; yield: 64%. Anal. calcd. (found) % of $C_{18}H_{40}N_4RbCl$ (*m.w.*: 433.46): C, 49.88 (49.79); H, 9.30 (9.28); N, 12.93 (12.87). IR (KBr disc, cm$^{-1}$): ν(N–H), 3258m; ν(C–H), 2962m; ν($CH_3$), 1372s; ν(C–C), 1184s. Molar conductivity in DMSO: 22 ohm$^{-1}$ cm$^2$ mol$^{-1}$. Magnetic moment $\mu_{eff}$ (B.M.): Diamagnetic.

**[RbL$_B$(NCS)]:** Potassium thiocyanate (0.194 g, 2.0 mmol) and [RbL$_B$Cl] (0.433 g, 1.0 mmol) were suspended and dissolved, respectively, in 20 mL hot methanol. Both solutions were then mixed and the resulting mixture was refluxed for 6 h. The reaction mixture was filtered and the filtrate was allowed to stand for two days after concentrating to about 10 mL. A white product of [RbL$_B$(NCS)] was separated by filtration and washed with methanol and diethyl ether. Finally, the product was stored in a vacuum desiccator over silica gel.

**[RbL$_B$(NCS)]:** Colour: white; yield: 56%. Anal. calcd. (found) % of $C_{19}H_{40}N_5RbS$ (*m.w.*: 433.46): C, 49.88 (49.79); H, 9.30 (9.21); N, 12.93 (12.87). IR (KBr disc, cm$^{-1}$): ν(N–H), 3265sh; ν(C–H), 2964m; ν($CH_3$), 1385s; ν(C–C), 110m, ν(CN) 2056 s, ν(CS) 854 w, δ(NCS), 460 m. Molar conductivity in DMSO: 35 ohm$^{-1}$ cm$^2$ mol$^{-1}$. Magnetic moment $\mu_{eff}$ (B.M.): Diamagnetic.

**Antimicrobial activities:** Antibacterial and antifungal activities of ligand and its alkali metal(I) complexes have been investigated against selected Gram-positive and Gram-negative bacteria and selected fungi as per standard protocols [15].

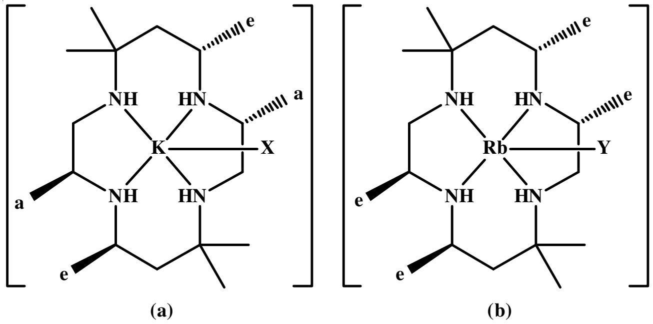
## RESULTS AND DISCUSSION

**Isomeric ligand $L_B$:** The octamethyl tetraazamacrocyclic isomer $L_B$ was synthesized and characterized following the protocols described elsewhere [14].

### Metal complexes

**Potassium(I) complexes:** Interaction between KX (Z = SCN, $NO_3$, Cl, Br and I) and octamethyl isomeric ligand $L_B$ in a methanolic solution with 11 h reflux in the ratio of 1:2 resulted in five coordinated square pyramidal white complexes [KL$_B$X]. The IR spectra of these complexes display ν(NH), ν(C–H), ν(C–C) and ν($CH_3$) bands, respectively, at 3253-3280, 2950-2982, 1142-1163 and 1350-1395 cm$^{-1}$. The further appearance of a sharp ν(CN) band at 2056 cm$^{-1}$ in the IR spectrum of [KL$_B$(NCS)] is a good indication of the N-bonded thiocyanate group [16,17]. However, it also displays ν(CS) band at 834 cm$^{-1}$ and δ(NCS) band at about 470 cm$^{-1}$ assigned for the fully N-bonded isothiocyanate group [17]. Moreover, the spectrum of [KL$_B$($NO_3$)] displays bands at 1437 and 1321 cm$^{-1}$ attributed to the coordinated $NO_3^-$ group. The separation of these bands by 116 cm$^{-1}$ accounted for a unidentate mode of coordination [18]. Moreover, the bands for KZ (Z = Cl, Br or I) expected at around 260 cm$^{-1}$ were undetected as the spectra could not be run below 400 cm$^{-1}$. The IR studies support the N-bonded thiocyanato complex and the unidentate mode of coordination by $NO_3$ group in the nitrato K(I) complex. Similar observation is also reported in the literature [19] for five coordinated square pyramidal K-complexes. Moreover, the molar conductivity value 22-39 ohm$^{-1}$ cm$^2$ mol$^{-1}$ of these complexes in DMSO corresponding to non-electrolyte [20] coincide with a square pyramidal structure.

The $^1$H NMR spectrum of [KL$_B$(NCS)] shows two singlets at δ 1.23 and 1.36 ppm corresponding to 6H each, originating from the two *gem*-dimethyl pairs. The spectrum further shows two doublets at δ 1.16 (6H) and 1.32 (6H) ppm, which can be assigned to two pairwise equivalent equatorial and two axial methyl groups on chiral carbon. The downfield multiplets may be due to methylene, methine and NH protons since these data support the diaxial and diequatorial orientation. Thus, based on the above studies, Fig. 1(a) can be assigned to K(I) complexes of $L_B$.



(a) (b)

Fig. 1. Structure of potassium complexes [KL$_B$(X)] (X = NCS, $NO_3$, Cl, Br and I) (a) and rubidium complexes [RbL$_B$(Y)] (Y = Cl and NCS) (b)

**Rb(I) complexes:** Interaction between Rb(I) chloride and $L_B$ in methanolic solution on 4 h reflux afforded five coordinated square pyramidal white complex, [RbL$_B$Cl]. However, [RbL$_B$Cl] underwent axial substitution reaction with KCNS in methanol in the ratio of 1:2 with 4 h reflux to furnish five coordinated square pyramidal white complex, [RbL$_B$(NCS)]. The IR spectra of these complexes display ν(NH), ν(C–H), ν(C–C) and ν($CH_3$) bands at 3265-3258, 2995-2962, 1184-1160 and 1385-1372 cm$^{-1}$, respectively. The bands for Rb–Cl expected at around 260 cm$^{-1}$ could not be detected, as the spectrum could not be run below 400 cm$^{-1}$. Moreover, the appearance of sharp ν(CN) band at 2056 cm$^{-1}$ for [RbL$_B$(NCS)] indicates the N-bonded thiocyanate group. The present complex also reveals the ν(CS) band at 854 cm$^{-1}$ and δ(NCS) band at about 460 cm$^{-1}$ assigned for fully N-bonded isothiocyanato complexes [17]. The molar conductance 22-35 ohm$^{-1}$ cm$^2$ mol$^{-1}$ in DMSO of Rb(I) complexes indicates that, both complexes are non-electrolyte as expected [20]. As per the above discussion, the molecular structure of these Rb(I) complexes of $L_B$ can be assigned as Fig. 1(b).

**Antimicrobial activities:** Antimicrobial activities of different macrocycles and their metal complexes have already been reported in the literature [4-8,21]. But, the same activities of macrocyclic complexes with alkali metal templates have not been reported. Hence, the antimicrobial potential of the newly synthesized alkali(I) metals of macrocyclic ligand $L_B$ has been studied. The antibacterial activities of the complexes were investigated against Gram-positive and Gram-negative bacteria, three of each. The antifungal activities of these compounds against four fungi were also performed in the same context.

**Antibacterial activities:** The K(I) and Rb(I) complexes of $L_B$ exhibit a varying range of activities, even though the ligand $L_B$ is inactive. Isothiacyanato (S1) and chlorido (S3) complexes of K(I) and isothiacyanato (S7) complexes of Rb(I) show antibacterial activities against all the studied bacteria. Nitrato complexes (S2) of K(I) against *S. aureus,* iodido complexes of K (S5) against *S. typhi* and *P. asaerginosa* and chlorido complexes of Rb(I) (S6) against all Gram-negative bacteria have not shown any noticeable activity. Moreover, isothiacyanato (S1) complex of K(I) against *S. aureus* and *E. coli*; nitrato complex (S2) of K(I) against *B. subtilis* and *S. typhi*; chlorido (S3) complex of K(I) against *B. subtilis* and *E. coli* showed remarkable activities. The complexes exhibited high activity values, although the concerned ligand and the non-coordinated metal salts showed zero or negligible activity (Table-1). The stability of these macrocyclic complexes prevents the liberation of ligands or metal ions in the test media [21]. Thus, the antibacterial activity shown by the complexes can be attributed to the alkali metal complexes themselves.

**Antifungal activities:** The *in vitro* evaluation of antifungal activities of isomeric ligand $L_B$, their alkali metal complexes, non-coordinated metal salts and standard fungicide, griseofulvin, have been studied in this work. Four selected fungi, *Aspergillus niger*, *Fusarium equiseti*, *Penicillium* and *Aspergillus flavus,* have been tested in this study. The tested complexes showed different activities against the fungi. The isothiacyanato (S1) complex of K(I) showed high activity against all fungi except penicillium. The bromido complex of K(I) (S4) against *F. equiseti* and the chlorido complex of Rb(I) (S6) against *F. equiseti* and *Penicillium* exhibited 100% activities. However, nitrato complex (S2) of K(I) against *F. equiseti*; halo complexes (S3, S4 and S5) of K(I) against *A. flavus* have not shown any activity (Table-2).

## Conclusion

Potassium and rubidium complexes of octamethyl isomeric ligand $L_B$ were synthesized by the direct interaction of K(I) and Rb(I) salts as well as by axial substitution reaction on an Rb(I) complex. In all cases, five coordinated square pyramidal complexes were presumed. Structures of the complexes have been assigned depending on the spectroscopic studies and the molar conductivity data. The electrolytic behaviour of the synth-

TABLE-1
ANTIBACTERIAL ACTIVITIES OF SYNTHESIZED ALKALI METAL COMPLEXES OF AN OCTAMETHYL ISOMERIC MACROCYCLE

| Sample code | Test compounds | Diameter of zone of inhibition (mm) (24 h incubation) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Gram-positive | | | Gram-negative | | |
| | | *S. aureus* | *B. cereus* | *B. subtilis* | *S. typhi* | *P. asaeruginosa* | *E. coli* |
| L1 | $L_B$ | 0 | 0 | 0 | 0 | 0 | 0 |
| S1 | $[KL_B(NCS)]$ | 17 | 10 | 12 | 12 | 11 | 22 |
| S2 | $[KL_B(NO_3)]$ | 0 | 12 | 18 | 18 | 16 | 11 |
| S3 | $[KL_BCl]$ | 10 | 10 | 16 | 12 | 14 | 16 |
| S4 | $[KL_BBr]$ | 16 | 12 | 12 | 10 | 10 | 9 |
| S5 | $[KL_BI]$ | 14 | 13 | 13 | 0 | 0 | 5 |
| S6 | $[RbL_BCl]$ | 12 | 11 | 10 | 0 | 0 | 0 |
| S7 | $[RbL_B(NCS)]$ | 11 | 10 | 12 | 12 | 14 | 11 |
| M1 | KCl | 0 | 2 | 2 | 5 | 4 | 3 |
| Control | DMSO | 0 | 0 | 0 | 0 | 0 | 0 |
| Standard | Ampicillin | 26 | 25 | 23 | 26 | 27 | 24 |

TABLE-2
ANTIFUNGAL ACTIVITIES OF SYNTHESIZED ALKALI METAL COMPLEXES OF AN OCTAMETHYL ISOMERIC MACROCYCLE

| Sample code | Test compounds | % of inhibition of mycelial growth | | | |
|---|---|---|---|---|---|
| | | *S. niger* | *F. equiseti* | *Penicillium* | *A. flavus* |
| L1 | $L_B$ | 20 | 35 | 25 | 22 |
| S1 | $[KL_B(NCS)]$ | 74 | 85 | 12 | 66 |
| S2 | $[KL_B(NO_3)]$ | 71 | 0 | 35 | 65 |
| S3 | $[KL_BCl]$ | 64 | 0 | 20 | 0 |
| S4 | $[KL_BBr]$ | 72 | 100 | 9 | 0 |
| S5 | $[KL_BI]$ | 45 | 0 | 15 | 0 |
| S6 | $[RbL_BCl]$ | 00 | 100 | 100 | 50 |
| M1 | $[RbL_B(NCS)]$ | 53 | 69 | 90 | 90 |
| M2 | KCl | 0 | 0 | 0 | 0 |
| Control | DMSO | 0 | 0 | 0 | 0 |
| Standard | Griseofulvin | 54 | 45 | 62 | 64 |

esized alkali metal complexes has been described using the molar conductivity data. The antibacterial and antifungal abilities of these complexes have been evaluated against various bacteria and fungi. Antimicrobial activity was high among the investigated alkali metal complexes. Antibacterial activities of some complexes are comparable to the standard compound, while antifungal activities were more significant than the standard.

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## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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