

Synthesis, characterization, electrochemical and antimicrobial properties of half-sandwich ruthenium(II) complexes containing substituted bipyridine ligands.

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Abstract: Half-sandwich ruthenium complexes, $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{N,N})\text{PF}_6]$ where (N,N) = 5,5-dimethyl-2,2'-bipyridine, 4,4'-di-tert-butyl-2,2'-bipyridine and 2,2'-bipyridine were prepared by reacting $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{Ru}(\mu\text{-Cl})\text{Cl}]_2$ dimer with the N,N'-bidentate ligands in a 1:2 ratio. Characterization of the complexes was done using ¹H NMR, elemental analyses, IR and UV/Vis spectroscopy. Electrochemical properties of the complexes were determined by cyclic voltammetry. Antimicrobial activities of the complexes and respective ligands were tested against drug resistant Gram-negative *Escherichia coli* ATCC 11775 and drug susceptible Gram-positive bacteria *Staphylococcus aureus* ATCC 12600. Some of the synthesized mononuclear ruthenium complexes demonstrated good potential antimicrobial activities against the selected bacteria tested with some showing better activity than well-known antibiotics such as streptomycin (S-10).

Keywords: Ruthenium, half-sandwich, antimicrobial susceptibility, electrochemical, bipyridine ligands.

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I. Introduction

The coordination chemistry and reactivity of half-sandwich ruthenium(II) complexes have attracted interest in the recent past because of their wide range application in alkene polymerization [1], catalysis [2,3,4], anticancer drugs [5], medicinal activities [6] and electrochemically active sensors [7]. The ruthenium(II) arene complexes present a three-legged piano stool structure through which the metal centre is in octahedral geometry and the arene ligand occupies the three coordination sites allowing the possibility to introduce other donor ligands which could influence its properties significantly [8]. These complexes have displayed significant antimicrobial properties and concerted research efforts to develop compounds that can counter resistance of bacteria to current range of drug [9, 10] is ongoing. For this reason synthesis of half sandwich ruthenium(II) complexes and their therapeutic application have been extensively studied. However no research has been undertaken to synthesize half-sandwich ruthenium complexes where the arene is toluene and using bipyridine ligands. Our interest is to synthesize new and noble ruthenium complexes, $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{N,N})\text{PF}_6]$ shown in Figure 1 using bipyridine ligands where (N,N) = 5,5-dimethyl-2,2'-bipyridine (1); 4,4'-di-tert-butyl-2,2'-bipyridine (2) and 2,2'-bipyridine (3), according to modified reported procedures [11]. Characterization of the new complexes, evaluation of their antimicrobial activity and their electrochemical properties are reported.

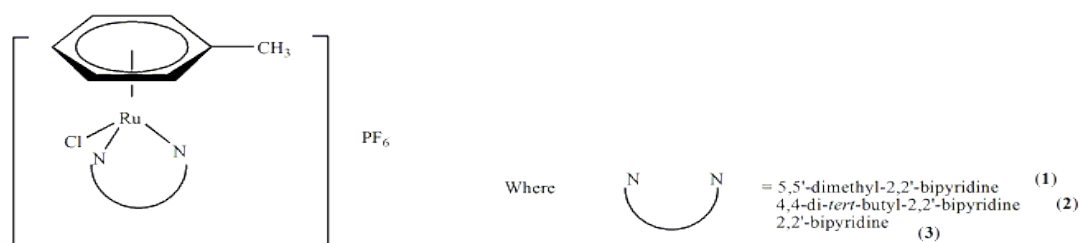


Figure 1: Half-sandwich ruthenium(II); $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{N,N})\text{PF}_6]$ complexes

II. Materials and Methods

All manipulations were carried out using modified Schlenk techniques under an inert atmosphere of nitrogen gas. Chemical reagents and solvents were obtained from the suppliers (Aldrich) and were used without further purification. Melting points were determined using a Gallenkamp Melting point apparatus. Elemental analyses were performed on a Vario EL III elemental CHNS/O analyzer. FT-IR spectra were recorded using an

ATR Perkin Elmer Spectrum 100 spectrophotometer. Electronic spectra were recorded in acetonitrile with a Perkin-Elmer Lambda 35 UV–visible spectrophotometer. ^1H NMR spectra were recorded on a Bruker Top Spin 400 MHz spectrometers using deuterated DMSO- d_6 . Electrochemical measurements were performed using BASi Epsilon E2 1177 model potentiostat, using a conventional three-electrode cell with platinum working electrode, platinum wire counter electrodes and a Ag/AgCl reference electrode.

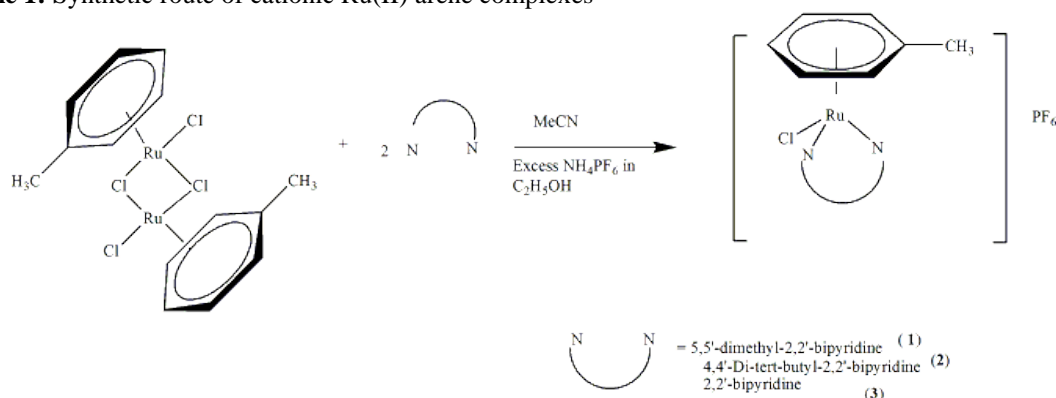
III. Experimental

Synthesis of monometallic half-sandwich $\text{N,N}'$ -bidentate Ruthenium complexes

$[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{N,N})\text{PF}_6]$ where $[\text{N-N} = \text{bipyridine and substituted bipyridine bidentate ligands}]$

The bimetallic arene precursor $(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{Ru}(\mu\text{-Cl})\text{Cl}_2$ synthesized using literature procedures [12] reacted with the N,N -bidentate ligands in dry acetonitrile at ambient temperatures to give new mononuclear half-sandwich complexes $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{N,N})\text{PF}_6]$, **1-3** as illustrated in Scheme 1 below.

Scheme 1: Synthetic route of cationic Ru(II) arene complexes



General synthetic procedures

Complexes **1–3** were synthesized following similar procedures. To a solution of the precursor complex $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{Ru}(\mu\text{-Cl})\text{Cl}_2]$ (106 mg, 0.20 mmol), dissolved in acetonitrile (10 cm^3) was added a slightly excess 5,5'-dimethyl-2,2'-bipyridine (0.43mmol, 79.22 mg) of the ligand dissolved in 15 mL acetonitrile for complex **1**, excess 4,4'-di-*tert*-butyl-2,2'-bipyridine (115.24 mg, 0.43mmol) for complex **2** and excess 2,2'-bipyridine (67.16 mg, 0.43mmol) for complex **3**. The mixture was stirred for 3 h under inert nitrogen atmosphere on a magnetic stirrer. Excess ammonium hexafluorophosphate (70.09 mg, 0.43 mmol) dissolved in ethanol (15 ml) was added to the yellow residue and the mixture cooled in an ice bath while stirring. The formed residue was filtered by vacuum filtration and washed twice with diethyl ether (10 ml) and dried in vacuo for 4 h.

Synthesis of $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{C}_{12}\text{H}_{12}\text{N}_2)]\text{PF}_6$ (**1**) :

Yield: (94.7 mg, 89 %). m.p $223\text{--}224^\circ\text{C}$; ^1H NMR (400 MHz, DMSO- d_6 , 295 K): $\delta = 9.37$ (s, 2H; Py), 8.44 (d, 2H; Py), 8.07 (d, 2H; Py), 6.32 (d, 2H, Ar), 5.94 (d, 2H, Ar), 5.78 (s, 1H; Ar), 2.51 (s, 6H, $(\text{CH}_3)_2$, Py), 2.23 (s, 3H, CH_3). (IR; KBr, cm^{-1}): 2927 (C-H)_{str}, 1608 (C=N), 1527 (C=C), 555 $\nu(\text{P-F}_6)$. Anal. Calculated: for $[\text{C}_{19}\text{H}_{20}\text{ClN}_2\text{Ru}]\text{PF}_6$: C, 40.93; H, 3.59; N, 5.02. Found: C, 40.91; H, 3.61; N, 5.02.

Synthesis of $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{C}_{18}\text{H}_{24}\text{N}_2)]\text{PF}_6$ (**2**) :

Yield: (96.5 mg, 90 %). m.p $227\text{--}228^\circ\text{C}$; ^1H NMR (400 MHz, DMSO- d_6 , 295 K): $\delta = 9.40$ (d, 2H; Py), 8.63 (d, 2H; Py), 7.70 (dd, 2H; Py), 6.25 (dd, 2H, Ar), 5.92 (d, 2H, Ar), 5.78 (s, 1H; Ar), 2.22 (s, 3H, $(\text{CH}_3)_3$, Ar), 1.43 (s, 18H, *tert*-butyl). (IR; KBr, cm^{-1}): 2971 (C-H)_{str}, 1619 (C=N), 1524 (C=C), 555 $\nu(\text{P-F}_6)$. Anal. Calculated: for $[\text{C}_{25}\text{H}_{32}\text{ClN}_2\text{Ru}\cdot\text{H}_2\text{O}]\text{PF}_6$: C, 45.49; H, 5.15; N, 4.24. Found: C, 44.83; H, 5.19; N, 4.24.

Synthesis of $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{C}_{10}\text{H}_8\text{N}_2)]\text{PF}_6$ (**3**) :

Yield: (95.8 mg, 90 %). m.p $210\text{--}211^\circ\text{C}$; ^1H NMR (400 MHz, DMSO- d_6 , 295 K): $\delta = 9.55$ (d, 2H; Py), 8.62 (d, 2H; Py), 8.27 (d, 2H; Py), 7.77 (s, 2H, Py), 6.30 (dd, 2H, Ar), 5.94 (d, 2H; Ar), 5.84 (s, 1H; Ar), 2.23 (s, 3H, Ar), ^{13}C NMR (400 MHz, DMSO- d_6 , 295 K): at 155.81(Py), 154.53(Py), 139.82(Py), 127.35(Py), and 123.65(Py), at 105.87(Ar) 90.53(Ar), 82.94(Ar), 79.73(Ar) 18.75(Ar). (IR; KBr, cm^{-1}): 3086 (C-H)_{str}, 1608 (C=N), 1527 (C=C), 555 $\nu(\text{P-F}_6)$. Anal. Calculated for $[\text{C}_{17}\text{H}_{16}\text{ClN}_2\text{Ru}\cdot\text{H}_2\text{O}]\text{PF}_6$: C, 37.25; H, 3.28; N, 5.11. Found: C, 37.38; H, 4.23; N, 5.51. ES/MS (M/Z): 385.0061 calculated for $[\text{C}_{17}\text{H}_{16}\text{ClN}_2\text{Ru}]^+$

IV. Results and Discussion

Physico-chemical characteristics of the cationic Ru(II)-arene complexes

Complexes **1-3** were obtained in good yields, they were air stable and readily soluble in polar solvents such as acetone, acetonitrile, DMSO and insoluble in non-polar organic solvents such as hexane and diethyl ether. Elemental analysis shows complex **2** and **3** may have retained water molecules trapped within the complex matrix which could not be removed during the drying stage. Table 1 shows summarized physico-chemical properties of the new complexes.

Table 1: Physico-chemical properties of $[(\eta^6\text{-arene})\text{RuCl}(\text{N,N})\text{PF}_6]$ complexes

COMPLEX	COLOUR	M.P	YIELD (%)	Molecular formulae	Calculated (Found)		
					C	H	N
1	Yellow	224	89	$[\text{C}_{19}\text{H}_{20}\text{ClN}_2\text{Ru}] \text{PF}_6$	40.93 (40.91)	3.59 (3.61)	5.02 (5.02)
2	Yellow	228	90	$[\text{C}_{25}\text{H}_{32}\text{ClN}_2\text{Ru}] \text{PF}_6 \cdot \text{H}_2\text{O}$	45.89 (44.83)	5.15 (5.19)	4.24 (4.24)
3	Yellow	211	90	$[\text{C}_{17}\text{H}_{16}\text{ClN}_2\text{Ru}] \text{PF}_6 \cdot \text{H}_2\text{O}$	37.25 (37.38)	3.28 (4.23)	4.23 (5.51)

(**1**) = $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{C}_{12}\text{H}_{12}\text{N}_2)]\text{PF}_6$; (**2**) = $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{C}_{18}\text{H}_{24}\text{N}_2)]\text{PF}_6$; (**3**) = $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{C}_{10}\text{H}_8\text{N}_2)]\text{PF}_6$

Electro-spray mass spectral analysis

The electro spray mass spectra of the complexes did not show the parent molecular ion (M^+) peak. However the most abundant ion peak in all the spectra was that of the fragment ion $[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{Ru}(\text{Cl})\text{bpy}]^+$. Which occur due to the loss of a PF_6 anion. A typical example of ES-MS of complex **3** is shown in Figure 2.

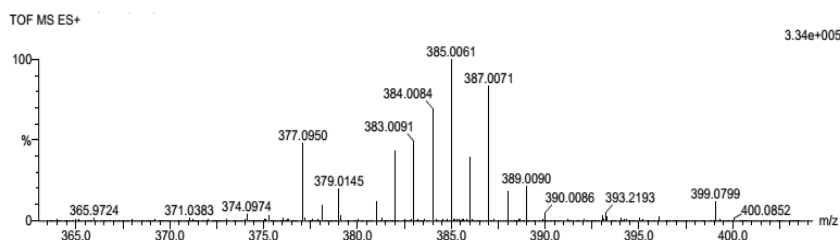


Figure 2: ES-MS spectrum showing the fragmentation pattern of a typical Ru(II)-arene complex.

Electronic spectral studies

Complexes **1-3** exhibit absorption bands of high intensity observed from 237 to 311 nm and of medium intensity from 317 to 344 nm attributed to the ligand centered $n\text{-}\pi^*$ and $\pi\text{-}\pi^*$ transitions [13] respectively in agreement with observations for complexes with similar N,N' bidentate ligands [14]. The complexes presented bands of low intensity at around 410 - 416 nm which may be attributed to the metal to ligand ($d\pi\text{-}\pi^*$) charge transfer (MLCT) transition from the filled metal 4d orbital to the empty π^* orbital as a result of the low-spin d^6 configuration of the Ru(II) complexes. This observations is in agreement with those made by the other authors [15]. Electronic spectra of complexes **1-3** are shown in Figure 3. Summarized electronic spectral data shown in Table 2.

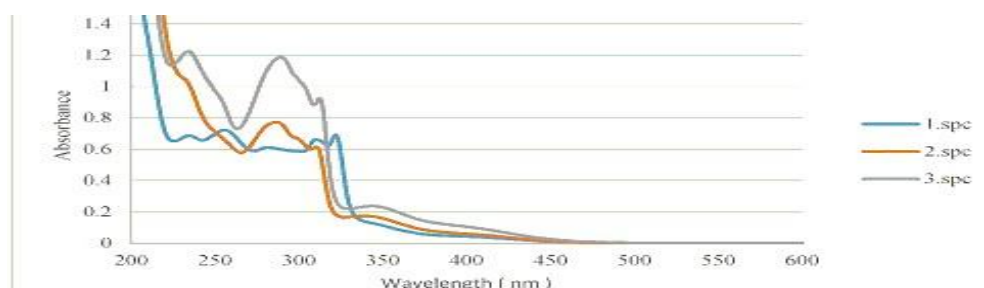


Figure 3: Electronic spectra of Ru(II)-arene bipyridine complexes ligands

Table 2: Electronic data for synthesized Ru(II)- arene bipyridine complexes

Serial	complex	$\pi \rightarrow \pi^*$	$n \rightarrow \pi^*$	MLCT
1.	$[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{C}_{12}\text{H}_{12}\text{N}_2)] \text{PF}_6$	(237,263) nm	359 nm	416 nm
2.	$[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{C}_{18}\text{H}_{24}\text{N}_2)] \text{PF}_6$	290 nm	348 nm	410 nm
3.	$[(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{RuCl}(\text{C}_{10}\text{H}_8\text{N}_2)] \text{PF}_6$	237 nm	(311,344) nm	416 nm

Infrared spectral studies

The IR spectra of complexes **1-3** show absorption bands in the range of 1618 cm^{-1} to 1608 cm^{-1} , this can be attributed to the symmetrical vibration stretch of the ($\nu \text{C}=\text{N}$) bond in the complexes, which shifted to lower wavenumbers when compared to the uncoordinated pyridine-imine ligands (1626–1600 cm^{-1}), indicating complex formation. Bands are observed in the 1582 to 1303 cm^{-1} region, corresponding to the different stretching frequencies of the C=C and C=N bonds due to N,N- bidentate ligands in these complexes. Strong absorption bands at 555 cm^{-1} observed for all complexes are attributed to the stretching frequency (P–F) of the PF_6 counter ion [16].

^1H NMR spectral studies

Resonance patterns in the ^1H NMR spectra of the complexes confirms formation of complexes as formulated. There is a notable downfield shift of arene based protons upon the complexation with ligands which may be caused by electron back donation of the $\text{CH}=\text{N}$ donating back to the ruthenium metal.

Electrochemical properties of the complexes

The electrochemistry of the new complexes was investigated using cyclic voltammetry. The cyclic voltammograms of the complexes all display reversible oxidations associated with $\text{Ru(II)} \rightarrow \text{Ru(III)} + e^-$ i.e. d^5/d^6 Ru(III)/Ru(II) couple (Table 3). While all the oxidations are electrochemically reversible, complex **2** displays an oxidation peak that is irreversible at 1.1 V attributed probably to either $\text{Ru}^{\text{III}} \rightarrow \text{Ru}^{\text{IV}} + e^-$ process or may be associated with the oxidation of the bipyridine ligands attached to the Ru(II) metal centre as observed in many mononuclear ruthenium complexes [17,18,19]. A comparison of the electrochemistry of complexes reveals that while complex **1** and complex **3** show slightly more anodic Ru(II)/Ru(III) couple, a much less anodic shift in the Ru(II)/Ru(III) couple is observed for complex **2**. As expected in the π – donor chloride ligand coordinated to the metal centre results to cathodic shift of the Ru(II)/Ru(III) couples. A typical example of complex **2** shown in Figure 4.

Table 3: Redox potentials for Ruthenium monometallic complexes in acetonitrile

COMPLEX	E_p^{ai} (Volts)	E_p^c (Volts)	E_p^{aii} (Volts)	$E_{1/2}$ (Volts)	ΔE (mV)
$[\text{C}_{19}\text{H}_{20}\text{ClN}_2\text{Ru}] \text{PF}_6$ (1)	0.54	0.63		0.587	90
$[\text{C}_{25}\text{H}_{32}\text{ClN}_2\text{Ru}] \text{PF}_6$ (2)	0.471	0.376	1.108	0.424	95
$[\text{C}_{17}\text{H}_{16}\text{ClN}_2\text{Ru}] \text{PF}_6$ (3)	0.642	0.525		0.584	125

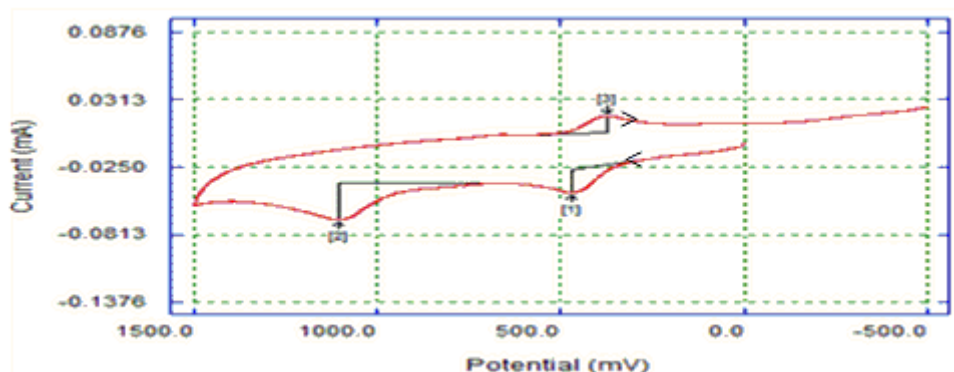


Figure 4: Cyclic voltammogram recorded at a platinum working electrode in CH_3CN solution of $[\text{C}_{25}\text{H}_{32}\text{ClN}_2\text{Ru}] \text{PF}_6$ complex **2** at a scan rate of 200 mV/s

Antimicrobial Susceptibility tests

All the synthesized complexes **1-3** were tested against some selected microorganisms to evaluate their antimicrobial activities. The disc diffusion method was used for antibacterial assay against the gram-positive bacteria *S.aureus* ATCC 12600 and the gram-negative bacteria, *E.coli* 11775. Streptomycin was used as standard drug for antibacterial screening and DMSO as the negative control [20]. The complex **1** $[\text{C}_{19}\text{H}_{20}\text{ClN}_2\text{Ru}] \text{PF}_6$ did not demonstrate any antimicrobial activity against both bacterial strains. Complex **2** was most effective against both bacterial strains while complex **3** showed no effectiveness against *S. aureus* but some activity was observed against *E.Coli*. Illustration shown in Figure 5.

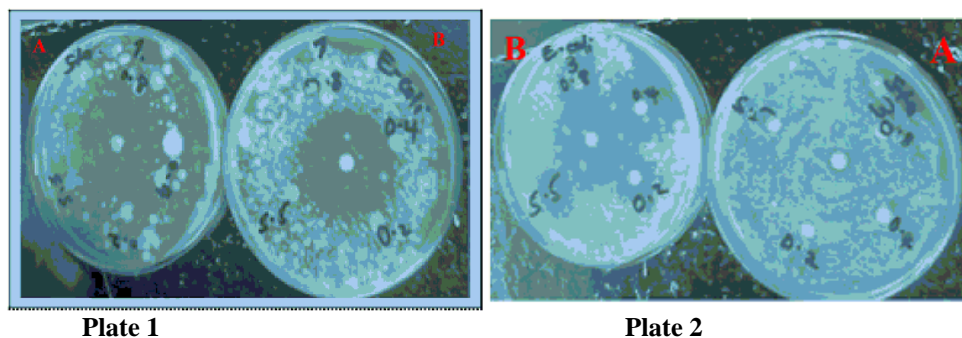


Figure 5. Antibacterial assay: Zone of inhibition against A) *E.coli* and B) *S.aureus* in plate 1 (complex 1) and plate 2 (complex 3)

V. Conclusion

The $[(\eta^6\text{-arene})\text{RuCl}(\text{N},\text{N})]\text{PF}_6$ complexes **1–3** were successfully synthesized from the reactions of $[(\eta^6\text{-arene})\text{Ru}(\mu\text{-Cl})\text{Cl}]_2$ dimers (where arene = toluene) with the N,N-bidentate ligands. The analytical data and spectral analysis reveal that the ligands acts as bidentate and coordinates to the Ru(II) metal ion through nitrogen. The complexes showed good antibacterial activity than free ligand against Gram positive bacteria, *Staphylococcus aureus* and Gram negative bacteria, *Escherichia coli*.

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