

Supplementary information

Unusual Electroluminescence in Ruthenium(II) Tetrazole Complexes

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S1:

The wavelength of electroluminescence (el) and photoluminescence (pl) emissions of reported ruthenium complexes.

Complex/reactant	λ_{em} (nm)	λ_{ecl} (nm)	ref
$Ru(bpy)_3^{2+}$	608	608	1,2,3
$Ru(bpy)_3^{2+}/C_2O_4^{2-}$	610	610	4
$Ru(bpy)_3^{2+}/C_2O_4^{2-}$		591	5
$Ru(bpy)_3^{2+}/S_2O_8^{2-}$	625	625	4,6
$Ru(bpy)_3^{2+}/TPrA$	610	610	7
$Ru(dmbp)_3^{2+}/C_2O_4^{2-}$		594	5
$Ru(phen)_3^{2+}$	590	590	8
$Ru(phen)_3^{2+}/C_2O_4^{2-}$		585	5
$Ru(dmphen)_3^{2+}/C_2O_4^{2-}$		591	5
$Ru(terpy)_3^{2+}$		660	8
$Ru(bpz)_3^{2+}$	585	585	9,10
$Ru(bpz)_3^{2+}/S_2O_8^{2-}$	585	590	11

$\text{Ru}(\text{dp-bpy})_3^{2+}$	635	635	12
$\text{Ru}(\text{dp-phen})_3^{2+}$	615	615	12
$(\text{bpy})_2\text{Ru}(\text{bphb})^{2+}$	624	624	13
$(\text{bpy})_2\text{Ru}(\text{bphb})^{2+} / \text{TPrA}$	624	624	13
$(\text{bpy})_2\text{Ru}(\text{bphb})^{2+} / \text{S}_2 \text{O}_8^{2-}$	624	624	13
$[(\text{bpy})_2\text{Ru}]_2(\text{bphb})^{4+}$	624	624	13
$[(\text{bpy})_2\text{Ru}]_2(\text{bphb})^{4+} / \text{TPrA}$	624	624	13
$[(\text{bpy})_2\text{Ru}]_2(\text{bphb})^{4+} / \text{S}_2 \text{O}_8^{2-}$	624	624	13
$(\text{bpy})_2\text{Ru}(\text{AZA-bpy})^{2+} / \text{TPrA}$	603	603	14
$(\text{bpy})_2\text{Ru}(\text{AZA-bpy})^{2+} / \text{TPrA}$	613	613	14
$(\text{bpy})_2\text{Ru}(\text{CE-bpy})^{2+} / \text{TPrA}$		650	15
$(\text{bpy})_2\text{Ru}(\text{CE-bpy})^{2+} / \text{TPrA}$		655	15
$\text{Ru}(\text{v-bpy})_3^{2+}$	630	650	16
$(\text{bpy})_2\text{Ru}(\text{DC-bpy})^{2+}$	629	629	17
$(\text{bpy})_2\text{Ru}(\text{DM-bpy})^{2+}$	605	605	17
$(\text{bpy})_2\text{Ru}(\text{dpen-bpy})^{2+} / \text{PF}_6^-$	612	612	18

$\text{Ru(m-bpy)}_3^{2+} / \text{PF}_6^-$	609	612	18
$\text{Ru(dtb-bpy)}_3^{2+} / \text{PF}_6^-$	610	611	18
$(\text{bpy})_2\text{Ru(DIM)}^{2+}$	600	600	19
$(\text{bpy})_2\text{Ru(PBIm-H)}^{2+} / \text{PF}_6^-$		680	20
$[\text{Ru(tpy)(tpy-COOEt)}] / \text{PF}_6^-$	706	706	21
Ru(DM-bpy)_3^{2+}	604	615	22
$(\text{bpy})_2\text{Ru(dbeb)}^{2+} / \text{PF}_6^-$	642	640	23
$(\text{bpy})_2\text{Ru(pbq)}^{2+}$	900	900	24
$(\text{PBIm-H})_2\text{Ru(pbq)}^{2+}$	945	945	24
$(\text{PBIm-H})_2\text{Ru(acac)}^{2+}$	850	880	24
$[\text{Ru(PBIM-H)}_2]_2(\text{pbq})^{+2}$	1040	1040	24
$\text{Ru(tpy)(trz)}^{2+} / \text{PF}_6^-$	723	717	25
$\text{Ru(tpy-COOEt)(trz)}^{2+} / \text{PF}_6^-$	717	725	25
$(\text{bpy})_2\text{Ru(Mt-bpy)}^{2+} / \text{PF}_6^-$	625	557	26

RuTRu	625	598	26
(bpy) ₂ Ru(aa-bpy) ²⁺ /PF ₆ ⁻	649	699	27
Ru ₂ (bpy) ₄ (im-phen) / ClO ₄ ⁻	638	655	28
(bpy) ₂ Ru(Eh-bpy) ²⁺ /PF ₆ ⁻	427	600	29
(bpy) ₂ Ru(Hmh-bpy) ²⁺ /PF ₆ ⁻	427	600	29
(H2MPy3,4DMPP)Ru(bpy) ₂ Cl /PF ₆ ⁻	655	656	30
Ru ₂ (bpy) ₂ (tpy) ₂ (BTB) ²⁺	680	710	31
Ru ₂ (bpy) ₂ (tpy) ₂ (4-TBN) ³⁺	676	680	31
[Ru(bpy) ₂] ₂ (bmpa-bpy) ⁺² / PF ₆ ⁻	642	596	32
[Ru(bpy) ₂] ₂ (bmdpa-bpy) ⁺² / PF ₆ ⁻	638	570	32
[Ru(bpy) ₂] ₂ (bmna-bpy) ⁺² / PF ₆ ⁻	636	570	32

m-bpy = 4-methyl-2,2'-bipyridine

dtb-bpy = 4,4'-di-tert-butyl-2,2'-bipyridine

dpen-bpy = 4,4'-di-n-pentyl-2,2'-bipyridine

DIM = 4,7-dimethyl-1,10-phenanthroline

PBI_m-H = 2-(2-pyridyl)-1H-benzoimidazole

tpy = 2,2',6',2''-terpyridine

tpy-COOEt = 2,2',6',2'', terpyridine-4'-carboxylic acid ethyl ester

DM-bpy = 4,4'-dimethyl-2,2'- bipyridine

dbeb= 4,4'-dibutyl ester-2,2'-bipyridine

pbq=2,3-bis(2-pyridyl)benzoquinoline

acac=acetylacetone

trz= 2-phenyl-4,6-dipyridin-2-yl-1,3,5-triazine

RuTRu = bis-2,2'-bipyridyl-ruthenium-bis-[2-((E)-4'-methyl-2,2'-bipyridinyl-4)-ethenyl]-thienyl-bis-2,2'-bipyridyl-ruthenium tetra hexafluorophosphate

Mt-bpy =4-methyl-4'-(2-thienylethenyl)-2,2'-bipyridine

aa-bpy= Acrylic acid 4'-acryloyloxymethyl-2,2'-bipyridinyl-4-ylmethyl ester

im-phen =1,2-bis(4-(1H-imidazo[4,5-f][1,10]phenanthroline-2-yl)phenoxy)ethane

Eh-bpy =4,4'-bis(3-ethylheptyl)-2,2'-bipyridine

Hmh-bpy =4-dihexylmethyl-4'-heptyl-2,2'-bipyridine

H2MPy3,4DMPP = meso-tris-3,4-dimethoxyphenyl-mono-(4-pyridyl)porphyrin

4-TBN = 4-(1H-tetrazol-5-yl)benzotrile

BTB = bis(1H-tetrazol-5-yl)benzene

bpy = 2,2'-bipyridine

C₂ O₄²⁻ = oxalate ion

S₂ O₈²⁻ = persulfate or peroxydisulfate

TPrA = tri-n-propylamine

dmbp = 4,4'-Me₂bpy and **DM-bpy** = 4,4'-dimethyl-2,2'-bipyridine

phen = 1,10-phenanthroline

terpy = 2,2',2''-terpyridine

bpz =2,2'-bipyrazine

dp-bpy = 4,4'-biphenyl-2,2'-bipyridyl

dp-phen =4,7-diphenyl-1,10-phenanthroline

dmphen = 4,7-dimethyl-1,10-phenanthroline

bphb = 1,4-bis(4'-methyl-2,2'-bipyridin-4-yl)benzene

AZA-bpy = 4-(N-aza-18-crown-6-methyl-2,2'-bipyridine

CE-bpy= bipyridine ligand where a crown ether (15-crown 5) is bound to the bpy ligand in the 3- and 3'-positions

v-bpy =4-vinyl-4'-methyl-2,2'-bipyridine

DC-bpy = 4,4'-dicarboxy-2,2'-bipyridine

PF₆⁻ =hexafluorophosphate

bmpa-bpy =bis(4'-methyl-2,2'-bipyridinyl-4-carbonyl)-(1,4-phenylenediamine)

bmdpa-bpy =bis(4'-methyl-2,2'-bipyridinyl-4-carbonyl)-(1,4-diphenylenediamine)

bmna-bpy =bis(4'-methyl-2,2'-bipyridinyl-4-carbonyl)-(1,4-naphthalenediamine)

S2:

2. Experimental :

2.1. Material and apparatus

All chemicals and solvents were purchased from Merck & Aldrich and used without further purification. IR spectra were recorded on a Perkin-Elmer 597 spectrometer ¹H-NMR spectra were recorded by use of a Bruker 250 MHz, spectrometer. The zirconium content of the final material was determined by ICP-AES model Perkin Elmer 1100DV. Electrochemical measurements were made in THF using model 273 A potentiostat. A conventional three-electrode configuration consisting of a glassy carbon working electrode, and Pt-wires as both the counter and reference electrodes was used. The supporting electrolyte was 0.1 M [Bu₄N]PF₆. Ferrocene was added as an internal standard after each set of measurements, and all potentials reported were quoted with reference to the ferrocene-ferrocenium (Fc/Fc⁺) couple at a scan rate of 100 mV/s. The oxidation (E_{ox}) and reduction (E_{red}) potentials were used to determine the HOMO and LUMO energy levels using the equations E_{HOMO} = - (E_{ox} + 4.8) eV and E_{LUMO} =

$-(E_{\text{red}} + 4.8)$ eV which were calculated using the internal standard ferrocene value of -4.8 eV with respect to the vacuum [33]. The PL spectra of the zirconium compounds and PVK:PBD were measured in 1,2-dimethylformamide solution. The PL spectra was recorded by ocean optic spectrometer USB2000 during 325 nm irradiation.

2.2. Synthesis of 5-Phenyl tetrazole ligand:

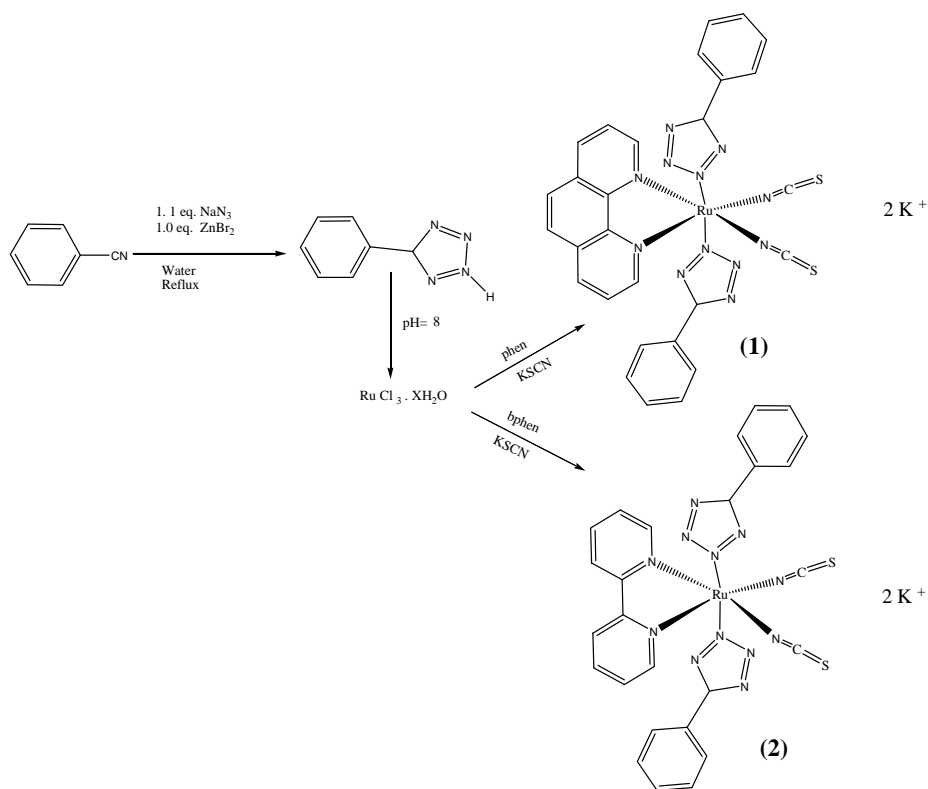
The 5-Phenyl tetrazole ligand was prepared according to the literature method [34]. benzonitrile (2.05 ml, 20 mmol), 40 ml of water, sodium azide (1.43 g, 22 mmol), and 4.5 g. (20 mmol) zinc chloride. The reaction was refluxed in a hood, but open to the atmosphere, for 24 h with vigorous stirring. After the mixture was cooled to room temperature, HCl (3 N, 30 mL) and ethyl acetate (100 mL) were added, and vigorous stirring was continued until no solid was present and the aqueous layer had a pH of 1. If necessary, additional ethyl acetate was added. The combined organic layers were evaporated, 200 mL of 0.25 N NaOH was added, and the mixture was stirred for 30 min, until the original precipitate was dissolved and a suspension of zinc hydroxide was formed. The suspension was filtered, and the solid washed with 20 mL of 1 N NaOH. To the filtrate was added 40 mL of 3 N HCl with vigorous stirring causing the tetrazole to precipitate. The tetrazole was filtered and washed with 2×20 mL of 3 N HCl and dried in a drying oven to furnish the tetrazole as a white or slightly colored powder.

2.3 Synthesis of complex (1):

A mixture of $\text{RuCl}_3 \cdot x\text{H}_2\text{O}$ (0.1 g, 1 mmol) and Tz (0.1409 g, 2mmol) in ethanol was refluxed for 30 min, then the solution of phen (0.0956 g, 1mmol) were added to the mixture to the refluxed for 30 min, then KSCN (0.0936g, 2mmol) was added, and the mixture was refluxed for 3h at 120 °C. After cooling down to the room temperature, the precipitate was filtered, washed with 2×20 mL ethanol, and dried in air . The obtained brown powders were soluble in DMF, acetonitrile and DMSO. Yield, 70% ;m.p. up250 °C.

2.4. Synthesis of complexe (2):

Tz (0.1409 g, 2mmol) dissolved in ethanol was added to a solution of $\text{RuCl}_3 \cdot x\text{H}_2\text{O}$ (0.1 g, 1 mmol) in ethanol (the pH value of the solution Tz was adjusted to 8 with solution NaOH) the reaction mixture was reflux for 1 hour, followed by a bpy(0.0753g, 1mmol) solution was added dropwise to the reaction mixture was reflux for 30 min and then a solution of KSCN(0.0936g, 2mmol) to above mixture was added and the reflux for 3 hours at 120°C . The black precipitate filtered and washed 2 times with 15 ml of ethanol and acetone. Yield, 74% ;m.p. up250 °C.



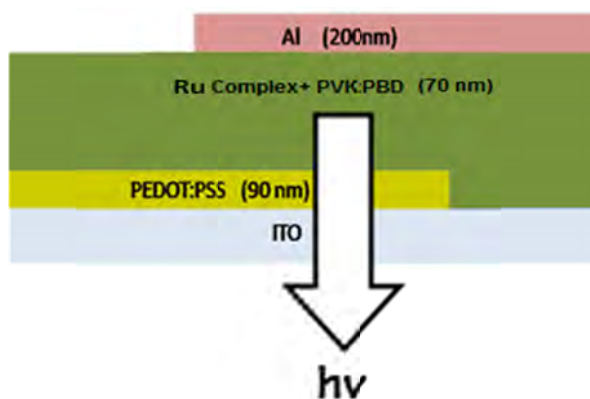
Scheme 1. The procedure of investigated Ru(Tz) complexes

2.5. Preparation of EL devices and testing

The structure of the fabricated device is as follow:

ITO/PEDOT:PSS(90nm)/PVK:PBD(70nm)/Al(200nm)and,

ITO/PEDOT:PSS(90nm)/PVK:PBD: ruthenium complex (70nm)/Al(200nm), That is shown in figure 1.



FigS2. the layer arrangement of ruthenium-based LED-device .

PVK as a hole-transporting and PBD as an electron-transporting material were doped with yttrium compounds. Glass substrates, coated with ITO (sheet resistance of $70 \Omega/\text{m}^2$), were used as the conducting anode. The ratio of ruthenium complexes for each type were 10 %Wt in PVK:PBD(100:40). PEDOT:PSS(poly(3,4-ethylenedi-oxythiophene):poly(styrenesulfonate)) was used as a hole injection and transporting layer. All polymeric layers were successively deposited onto the ITO coated-glass by using spin-coating process from the solution. A metallic cathode of Al was deposited on the emissive layer at 8×10^{-5} mbar by thermal evaporation. The PEDOT:PSS was dissolved in DMF, spin coated on ITO and was held in an oven at 120 oC for 2 hours after deposition. PVK, PBD and yttrium complexes with ratio of 100:40:10 were blended in DMF, and then spin coated and baked at 80°C for 1 hour. The thickness of the polymeric thin film was determined by a Dektak 8000. The EL intensity and spectra were measured with an ocean optic USB2000, under ambient conditions. In addition, Keithley 2400 sourcemeter was used to measure the electrical characteristics of the devices.

Results and Discussion

S3

IR characterization:

The IR spectra of complexes were found to displace from that of the ligand, the multiple vibrational bands due to N-H stretching's observed in the free Tz in the 2600-3100 cm^{-1} region disappeared [35]. Absorption band of Tz ring C-N and phenyl ring C-H and C=C observed in 1161 cm^{-1} , 1081 cm^{-1} and 3166 cm^{-1} and 1607 cm^{-1} , respectively [36]. IR spectra show absorption bands of C=N and N=N for the free Tz at 1484 cm^{-1} and 1409 cm^{-1} are shifted on complexation to lower frequencies 1445 and 1381 cm^{-1} (1) and 1445, 1365 cm^{-1} (2) respectively. These shifts could result from delocalization of the negative charge into the ring of the tetrazolate anion, which gives the ring a partial aromatic characterization.[37]. Moreover, significant changes are obvious in the region of stretching and stretching-deformation vibrations of the tetrazole ring (1100–900 cm^{-1}) [38]. The observed changes in absorption bands of endocyclic bonds of tetrazoles under the complexation could be explained by the electron density transfer from the tetrazole ring antibinding orbital to metal ion to form the bonds metal–N tetrazoleate, leading to a disruption of the aromaticity of the heterocycle as a whole [39]. Stretching frequencies C=N and C=S in the area 2050-2100 cm^{-1} the exhibit presence of ligand SCN^- in the complexes (1) and (2). The absorption band at 480 cm^{-1} attributes to the stretching vibration of Ru-N, showing that the phen or bpy are coordinated with Ru(II) ions by nitrogen moiety of phen or bpy, which are a strong evidence of the formation of the complex.

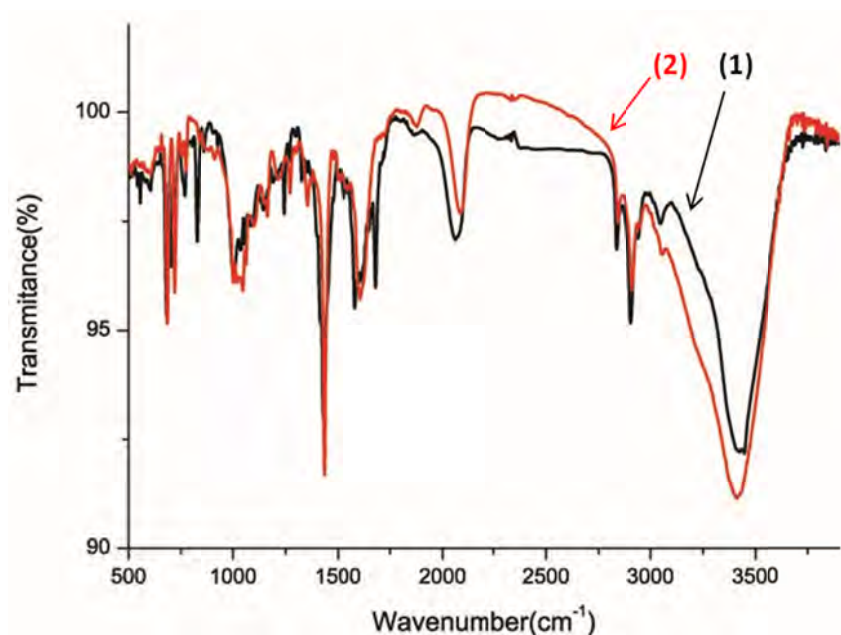


Fig. S3 FT-IR spectra of complexes (1) and (2).

S4

NMR characterization:

The spectrum of complex (1) contains signals of four distinguishable hydrogens of phen which some of them are overlapped by the hydrogens of Tz rings. For complexes (2), the occurrence of only four bpy signals confirms the presence of one compound. The downfield-shifted proton

resonance peaks can be assigned to the protons that are closed to the nitrogen of pyridine units, whereas the high-field proton resonances are assigned to the protons that are in the vicinity of the non-nitrogen atoms. With the use of Ru-phen it has been demonstrated that the metal ion having strong a back bonding capability causes an upfield shift of the proton signals of phen. Analogously, the upfield shift of bpy protons of Ru-bpy is explained by increased a back-bonding in Ru-bpy and concurrently decreased a back-bonding in Ru-L [40, 41]. The signal integration for complex (1) reveals the incorporation of one phen and two Tz units. For complexes (2), one bpy and two Tz units were found by signal integration.

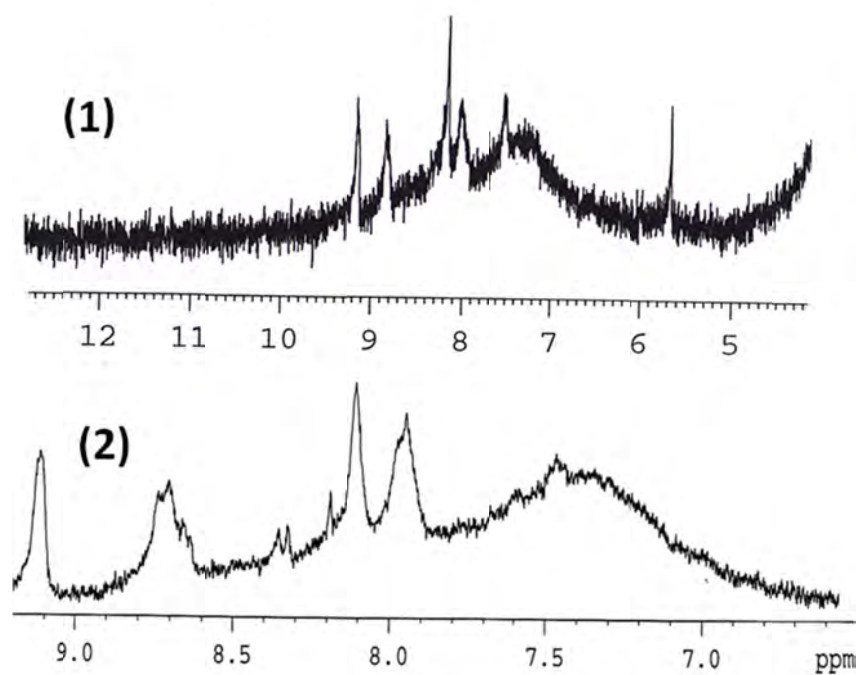


Fig. S4. The 250 MHz ¹H-NMR spectrum of complexes (1) and (2) recorded at 25°C in d-DMSO solvent.

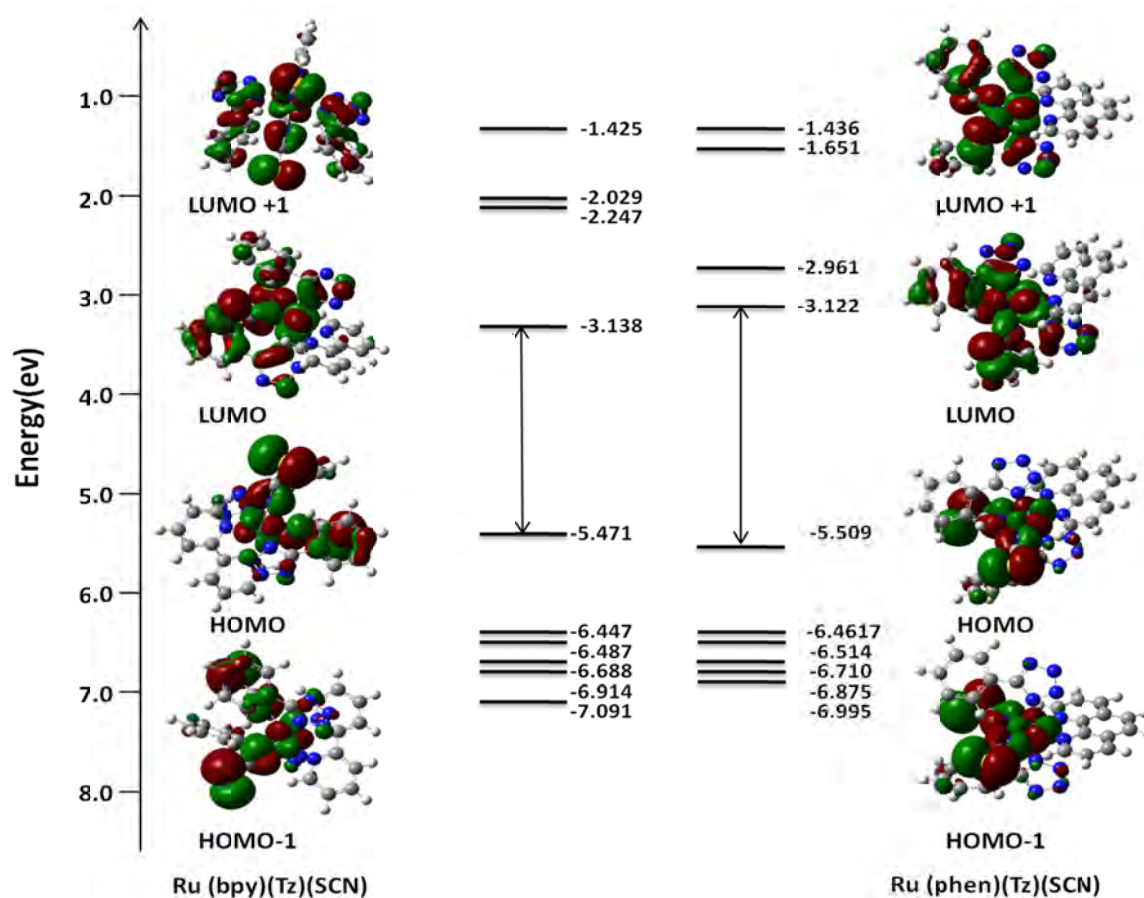
S5:

CHN and ICP analysis were carried out to determine the presence of ligands and metal in the complexes. CHN analyses of compounds were obtained: Anal. Calc. for (1) , (C₂₉H₁₈N₁₂S₂Ru) : C, 48.908; H, 2.620; N, 24.454. Found: C, 48.919; H, 2.598; N, 24.422%. Anal. Calc. for (2) , (C₂₆H₁₈N₁₂S₂Ru) : C, 47.058; H, 2.714; N, 25.339. Found: C, 47.102; H, 2.698; N, 25.287%. Ru was analyzed on a PLASMA-SPEC (I) ICP atomic emission spectrometer. A few mg of complexes (0.02g) were destroyed in HNO₃ (68%) and finally diluted in water to 1:10 for being measured. The found concentrations for all complexes were estimated about 3- 3.3 ppm.

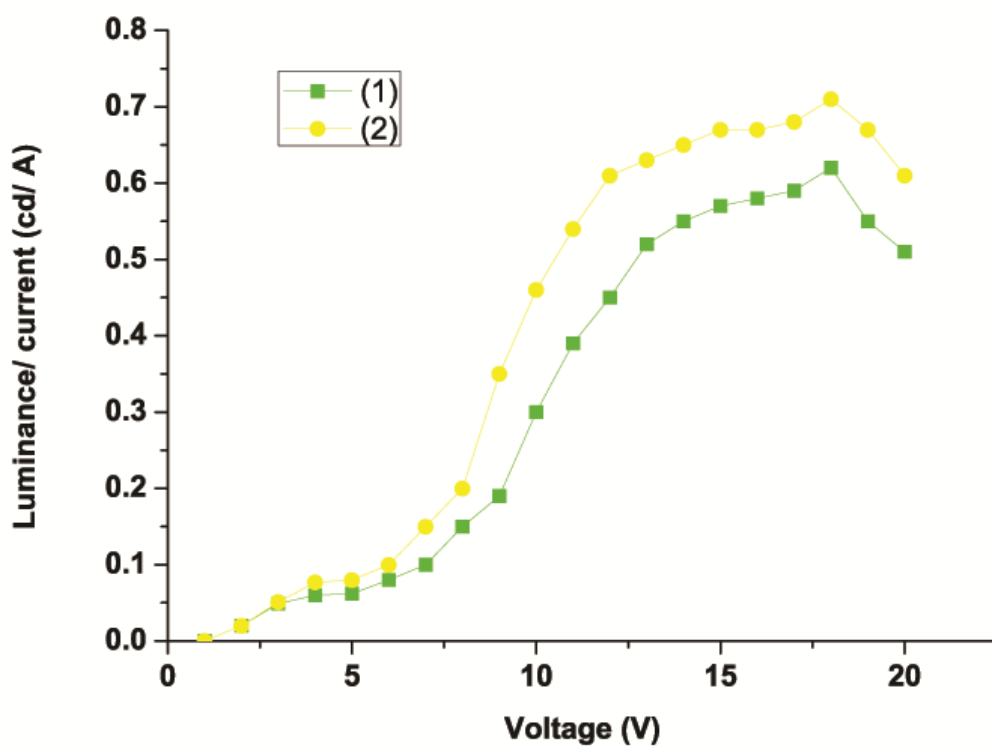
S6

To gain further insight into the electron density distribution within the frontier and other close lying orbitals, we performed quantum chemical calculations on the complexes (1) and (2) using density functional theory (DFT) at the GEN BASIS SET. The simulations have been performed with the Gaussian 09 program. The levels of scc-pVTZ and 6-31g++G(d,p) were carried out for atoms of SNO and CH, respectively.

Fig. S6. Electron density contours and energy values (in eV) calculated for the highest occupied (HOMO-4 to HOMO) and lowest unoccupied (LUMO to LUMO+4) molecular orbitals of complex (1) and (2) in the presence of the solvent (acetonitrile).



S7. The maximum efficiency (LE) versus applied voltages (V) characteristics of devices (1, 2).



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