New ruthenium(II) complexes with cyclic thio- and semicarbazone: Evaluation of cytotoxicity and effects on cell migration and apoptosis of lung cancer cells

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Supplementary Data

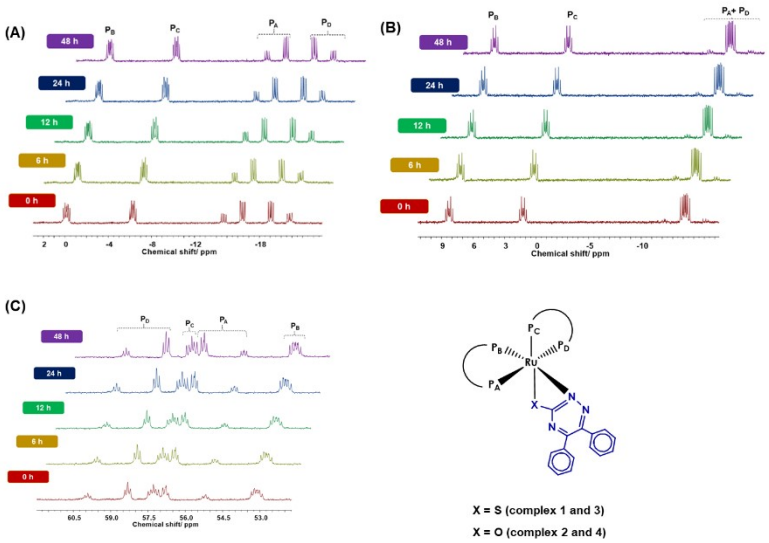

Figure S1. ^{31}P $\{^{1}H\}$ NMR spectra of complex (A) **1**, (B) **2** and (C) **4**, obtained at 0 (red line), 6 (yellow line), 12 (green line), 24 (blue line) and 48 h (purple line) after solubilization in the DMSO-d_6 solvent, with phosphorus atoms identified.

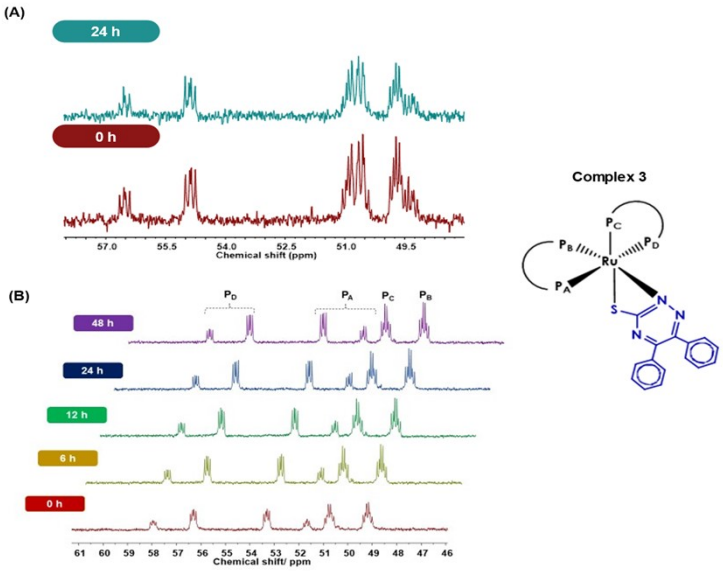

Figure S2. ^{31}P $\{^{1}H\}$ NMR spectra of complex **3** obtained at (A) 0 (dark red line) and 24 h (blue line) after solubilization in the PBS 1X, with 10% (w/v) of DMSO; (B) 0 (red line), 6 (yellow line), 12 (green line), 24 (blue line) and 48 h (purple line) after solubilization in the DMSO-d_6 solvent, with phosphorus atoms identified.

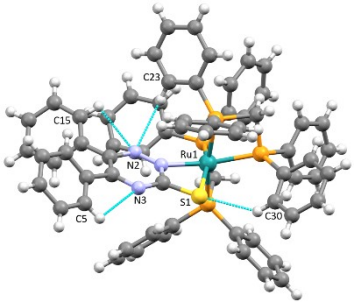

Figure S3. Intramolecular interactions, represented by the blue dotted lines, in the crystal packing of complex **1**·0.5CH_2Cl_2.

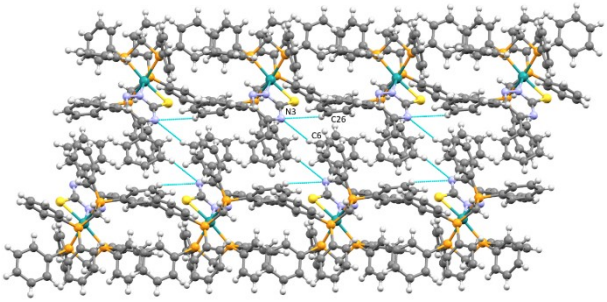

Figure S4. Intermolecular interactions, represented by the blue dotted lines, in the crystal packing of complex **1**·0.5CH_2Cl_2 that involves the N3 atom.

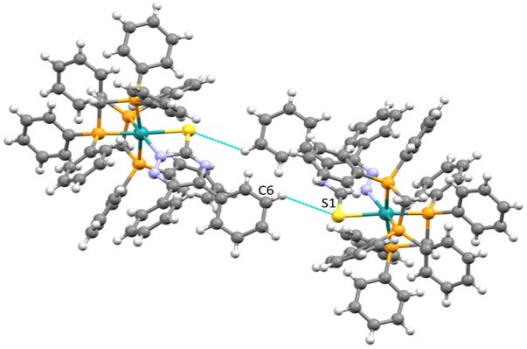

Figure S5. Intermolecular interactions, represented by the blue dotted lines, in the crystal packing of complex **1**·0.5CH_2Cl_2 that involves the S1 atom.

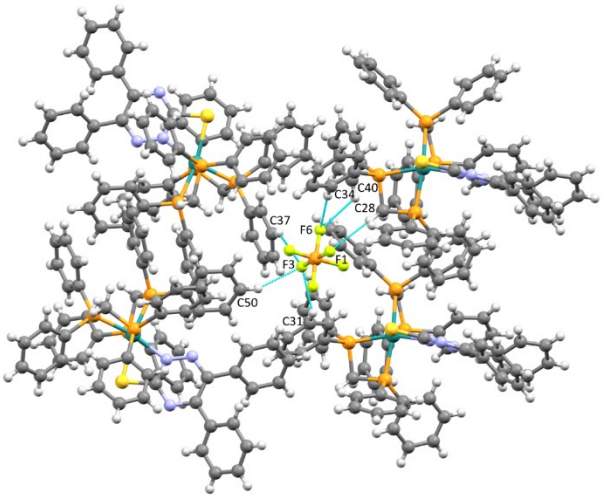

Figure S6. Intermolecular interactions, represented by the blue dotted lines, in the crystal packing of complex **1**·0.5CH_2Cl_2 that involves the PF_6^- counter ion.

Table S1. Crystal data and structure refinement for **1**·0.5CH_2Cl_2.

Empirical formula	$C_{65}H_{54}P_5SN_3F_6Ru·0.5CH_2Cl_2$	
Formula weight (g mol^{-1})	1321.55	
Temperature (K)	296(2)	
Wavelength (Å)	0.71073	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 20.283(2) Å	α = 90°
	b = 11.3236(10) Å	β = 104.824(4)°
	c = 29.017(3) Å	γ = 90°
Volume ($Å^3$)	6442.7(12)	
Z	4	
Absorption coefficient (mm^{-1})	0.458	
Crystal size (mm^3)	0.33 x 0.11 x 0.02	
Theta range for data collection (°)	1.452 a 26.365	
Index ranges	-25 ≤ h ≤ 25 -14 ≤ k ≤ 14 -36 ≤ l ≤ 36	
Reflections collected	159105	
Data/restraints/parameters	13109 / 155 / 856	
Absorption correction	Full matrix least-squares on F^2	
Goodness-of-fit on F^2	1.017	
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0505	

R indices (all data)	$wR_2 = 0.1116$ $R_1 = 0.0803$ $wR_2 = 0.1278$

Table S2. Main calculated distances for intermolecular interactions between 1-4 complexes and DNA.

Complex	Interaction nature	Interaction	Distance (Å)
(1)	Hydrophobic (π- π)	DG10···LIG1:dppm	4.47
		DG10···LIG1:dppm	3.39
		DG10···LIG1:dppm	4.39
		DG9···LIG1:dppm	3.67
		DG9···LIG1:dppm	4.49
		DC1···LIG1:dppm	5.65
	Hydrogen bond (S···H and S···O)	DG9:H21···LIG1:S	2.73
		DG9:H22···LIG1:S	2.51
		LIG1:S···DC2:O2	3.04
	π- S	LIG1:S···DG9	5.58
		LIG1:S···DG9	5.61
(2)	Hydrophobic (π- π)	DG9···LIG2:dppm	4.28
		DG9···LIG2:dppm	3.67
		DG10···LIG2:dppm	4.26
		DG10···LIG2:dppm	3.26
		DG10···LIG2:Bsc	4.74
		DC1···LIG2:Bsc	3.89
		DC1···LIG2:dppm	4.69
		DC2···LIG2:dppm	3.60
	Hydrogen bond (N···H)	DG10:H1'···LIG2:N	2.91
	Electrostatic (π- Anion)	DC2:OP1···LIG2:dppm	3.70
(3)	Hydrophobic (π- σ)	LIG3:C1···DG9	3.85
	Hydrogen bond (S···O and N···O)	LIG3:S···DC2:O2	3.30
		LIG3:N···DC2:O4'	3.00
(4)	Hydrophobic (π- π)	DC2···LIG4:Bsc	3.31
		DC1···LIG4:Bsc	4.33
		DC1···LIG4:Bsc	5.55
		DG9···LIG4:dppe	3.88
		DG9···LIG4:dppe	4.12
		DG10···LIG4:dppe	3.35

		DG10···LIG4:dppe	4.18
	Hydrogen bond (N···H and O···H)	DG9:H21···LIG4:N	2.50
		DG9:H22···LIG4:O	1.90

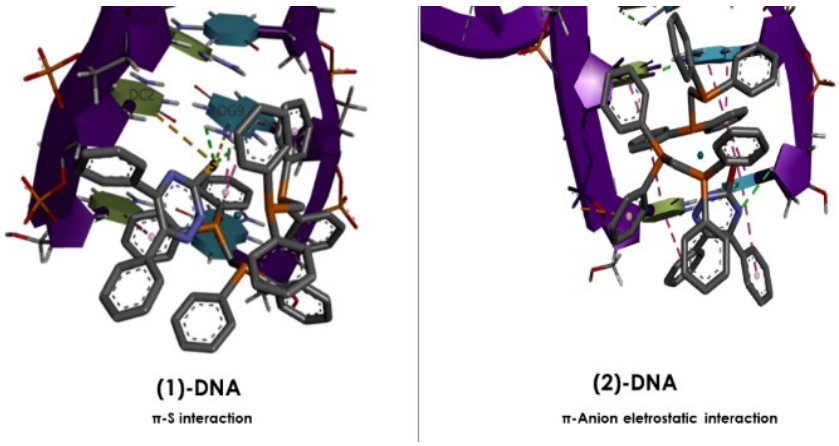

Figure S7. Other interactions suggested for complexes 1 and 2, π- S interaction (dashed yellow line) between complex 1 and DNA and π-Anion (dashed orange line) electrostatic interaction for complex 2 and DNA.

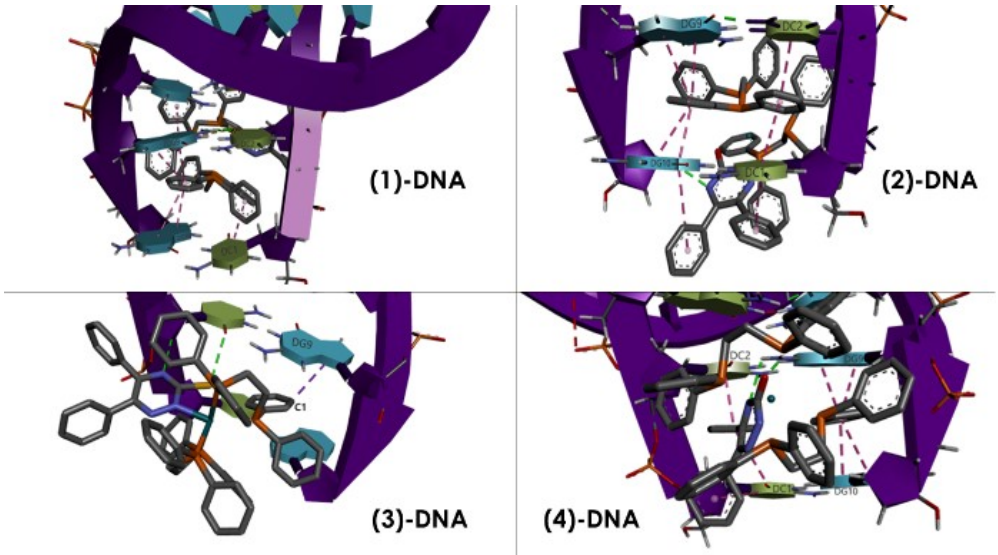

Figure S8. Hydrophobic interactions of π- π nature (dashed pink line) and π- σ (dashed purple line) for complexes 1-4 and DNA.

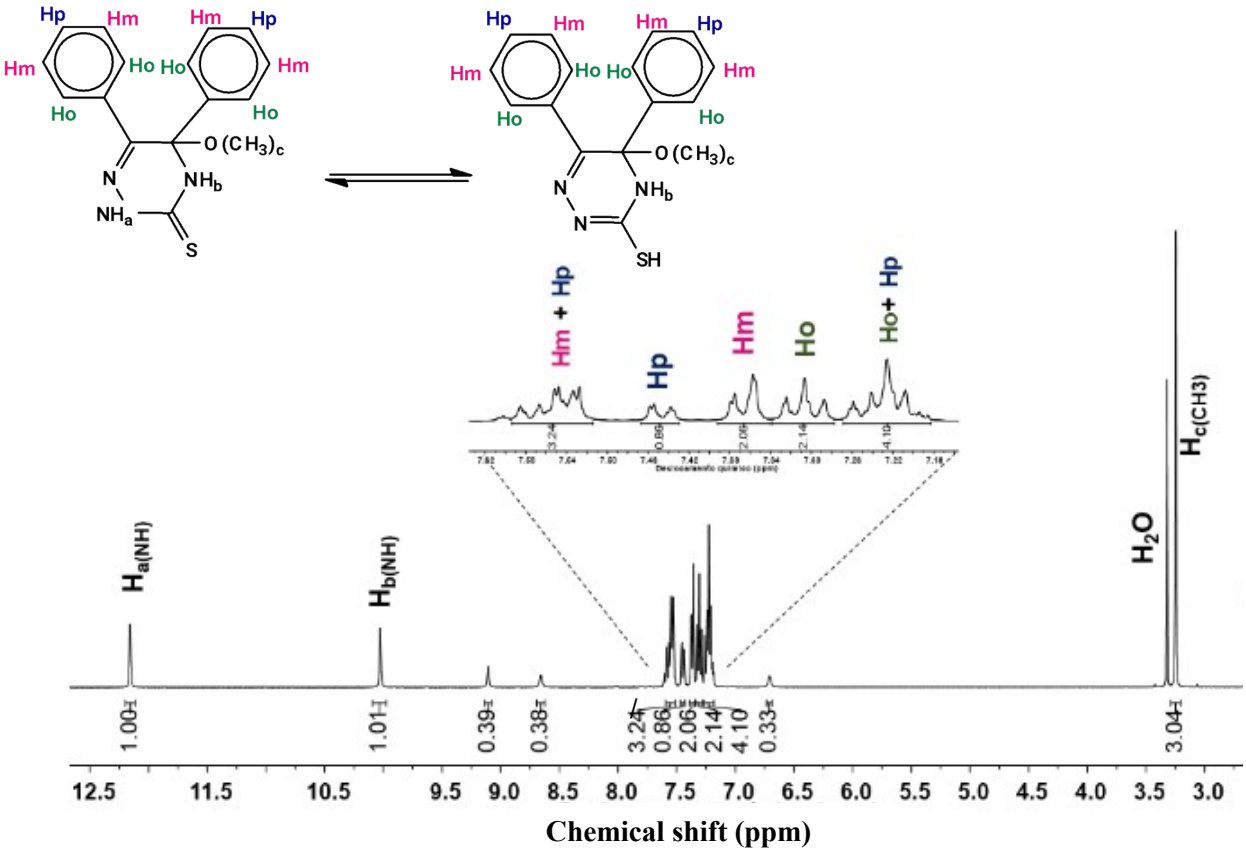

Figure S9. ^{1}H NMR spectrum of free ligand Btsc, obtained in d^{6}-DMSO and frequency of 400 MHz, with identified hydrogens and magnification in the region between 7.65–7.10 ppm.

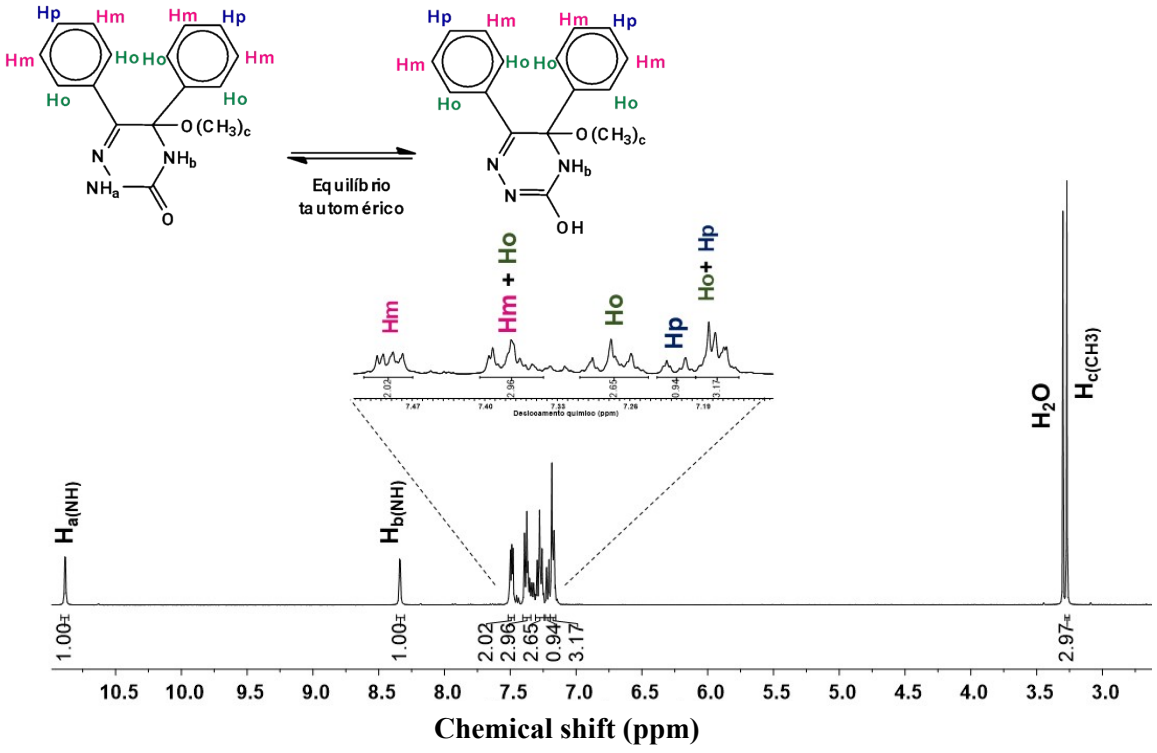

Figure S10. [1]H NMR spectrum of free ligand Btsc, obtained in DMSO-$d_6$and frequency of 400 MHz, with identified hydrogens and magnification in the region between 7.60–7.10 ppm.

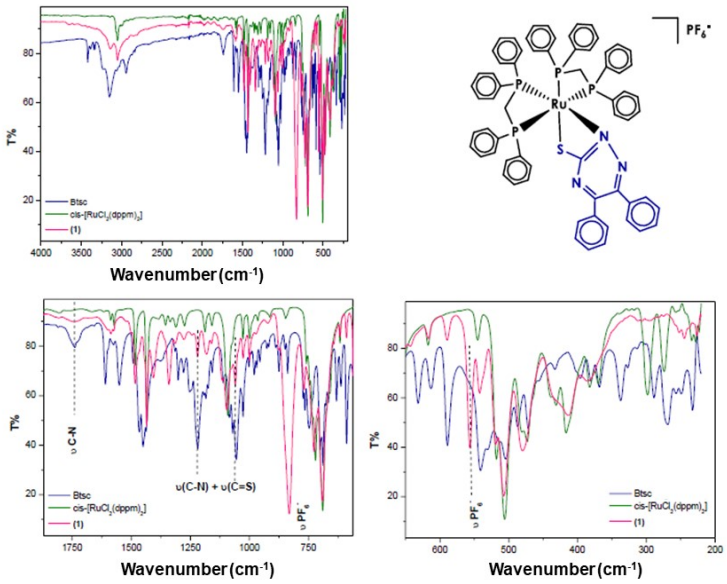

Figure S11. FTIR-ATR spectra of the Btsc free ligand (blue line), the cis-[$RuCl_2(dppm)_2$] precursor (green line) and complex **1** (pink line).

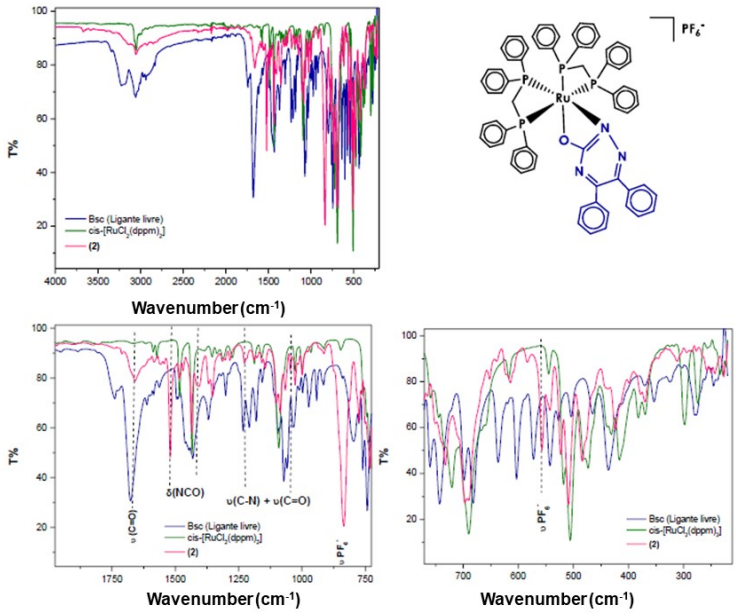

Figure S12. FTIR-ATR spectra of the Bsc free ligand (blue line), the cis-[$RuCl_2(dppm)_2$] precursor (green line) and complex **2** (pink line).

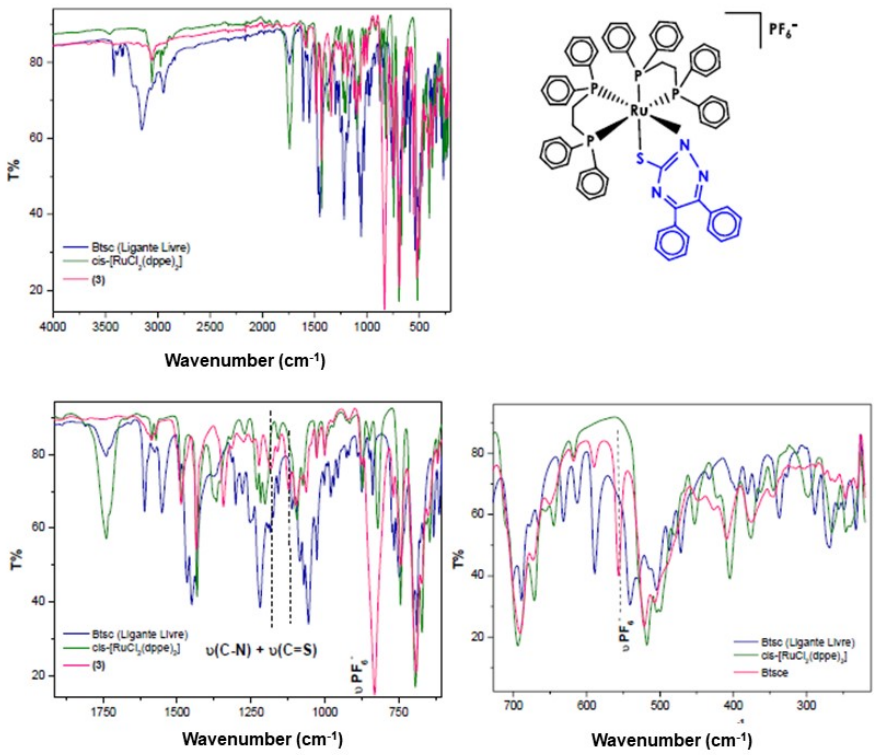

Figure S13. FTIR-ATR spectra of the Btsc free ligand (blue line), the cis-[$RuCl_2(dppe)_2$] precursor (green line) and complex **3** (pink line).

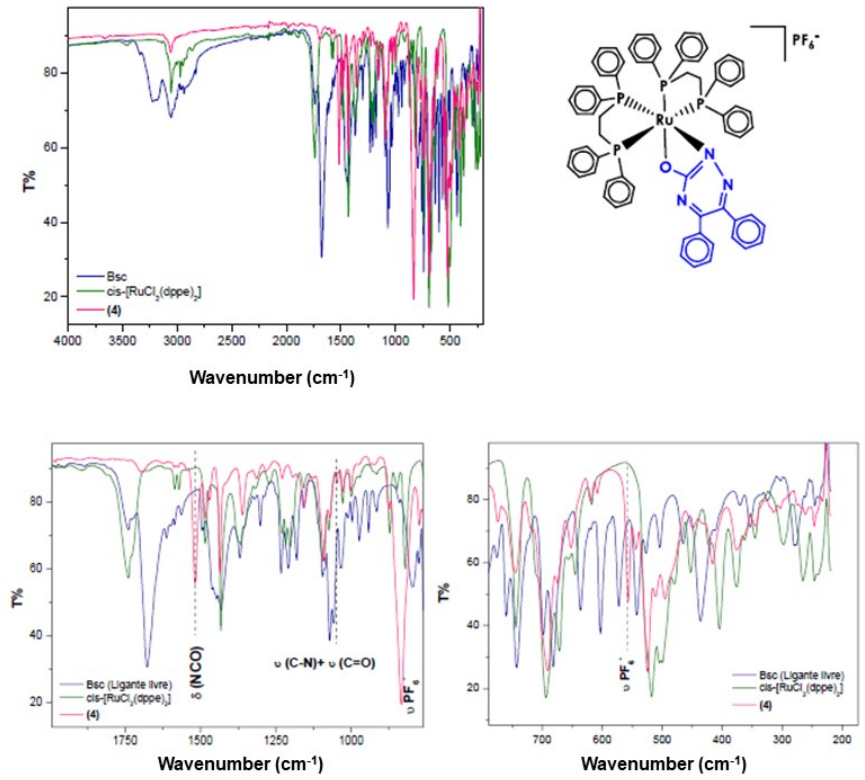

Figure S14. FTIR-ATR spectra of the Bsc free ligand (blue line), the cis-[$RuCl_2(dppe)_2$] precursor (green line) and complex **4** (pink line).

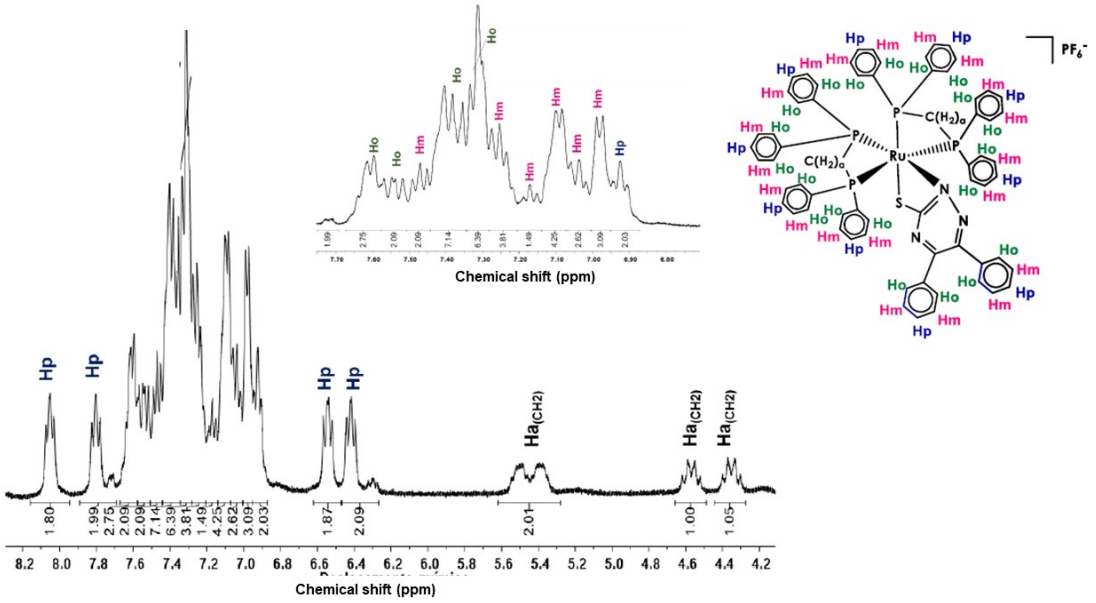

Figure S15. ^{1}H NMR spectra of complex **1**, obtained in DMSO-d_6 solvent at a frequency of 400 MHz, with its identified hydrogens.

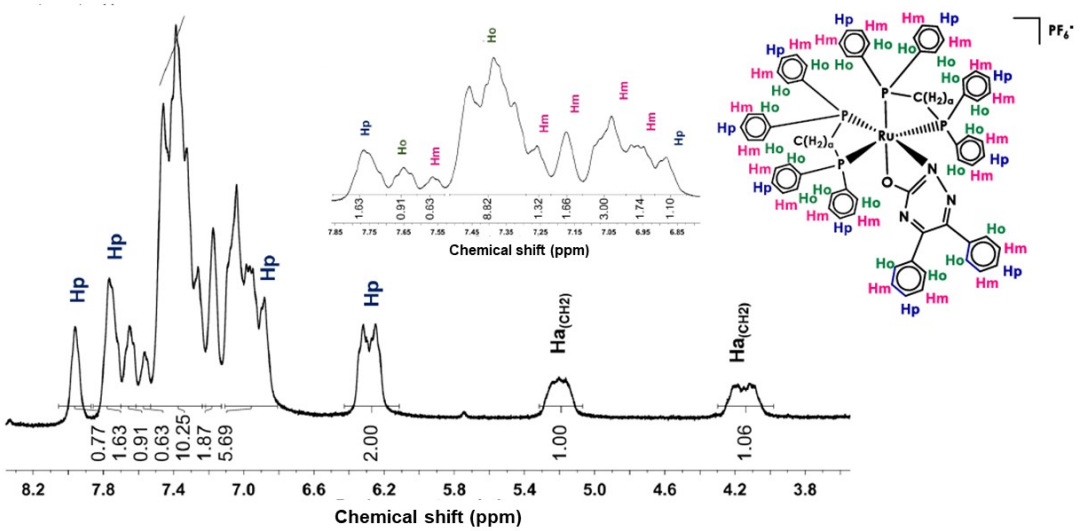

Figure S16. ^{1}H NMR spectra of complex **2**, obtained in DMSO-d_6 solvent at a frequency of 400 MHz, with its identified hydrogens.

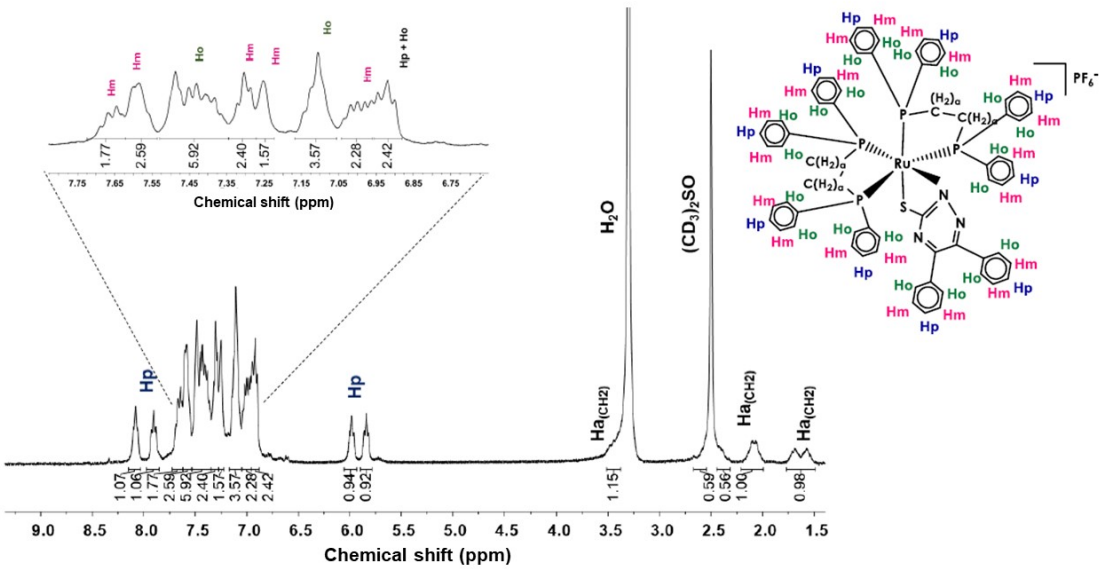

Figure S17. [1]H NMR spectra of complex **3**, obtained in DMSO-d_6 solvent at a frequency of 400 MHz, with its identified hydrogen atoms.

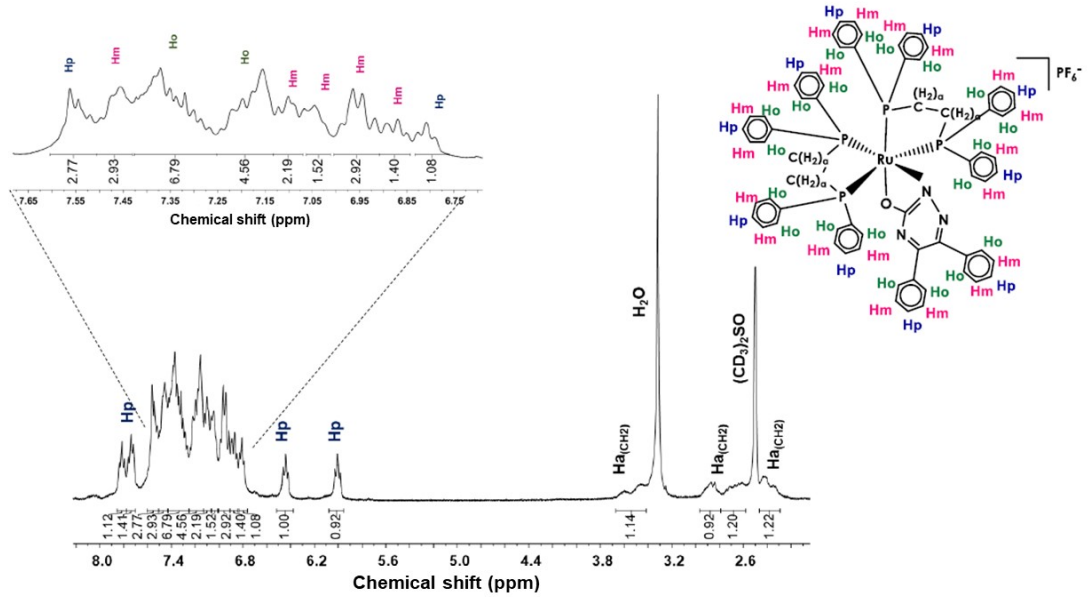

Figure S18. [1]H NMR spectra of complex **3**, obtained in DMSO-d_6 solvent at a frequency of 400 MHz, with its identified hydrogen atoms.

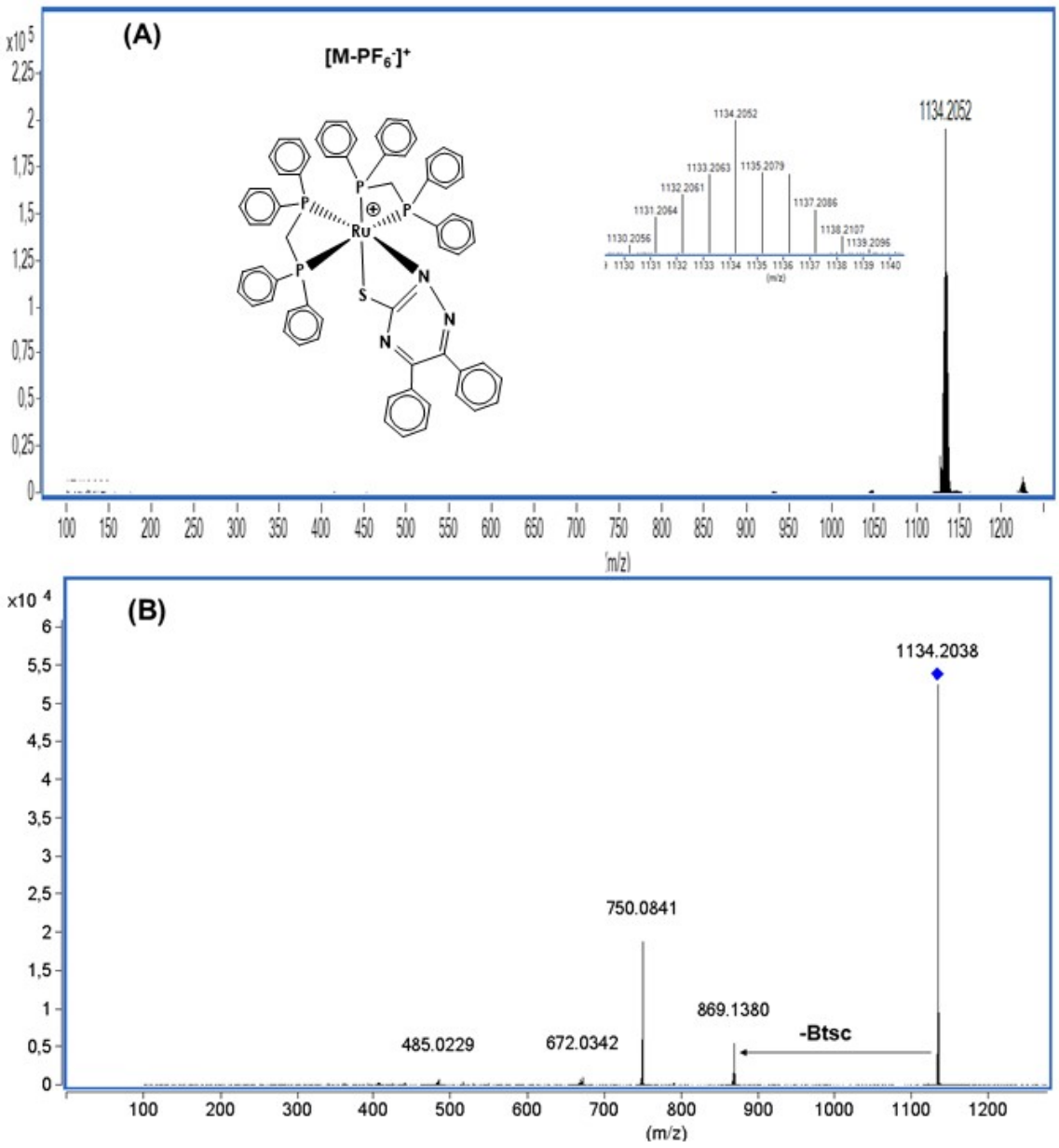

Figure S19. (A) HRESI-MS mass spectrum of complex **1. (B)** HRESI-MS/MS spectrum with fragmentation pattern of complex **1.**

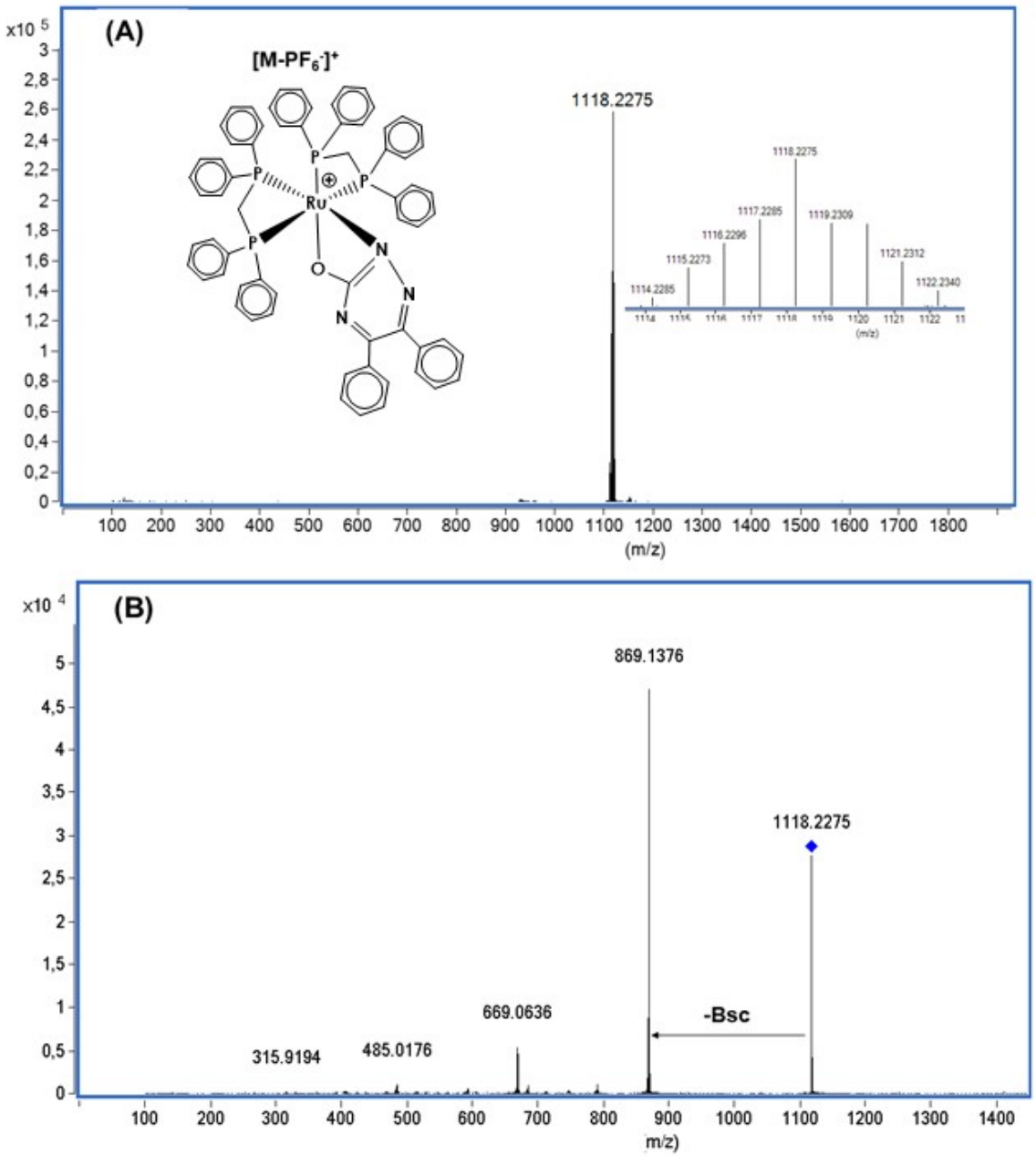

Figure S20. (A) HRESI-MS mass spectrum of complex **2. (B)** HRESI-MS/MS spectrum with fragmentation pattern of complex **2.**

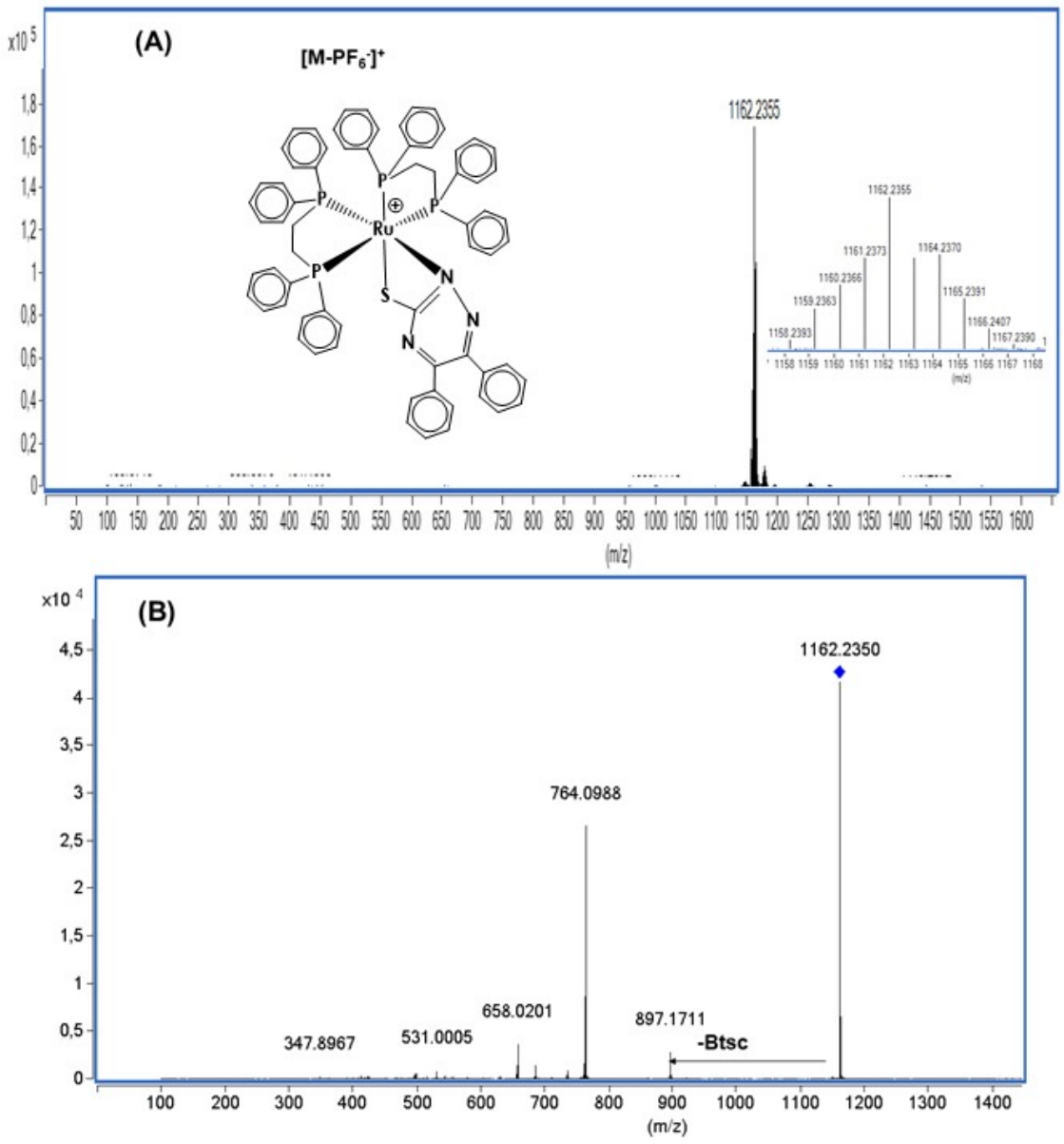

Figure S21. (A) HRESI-MS mass spectrum of complex **3. (B)** HRESI-MS/MS spectrum with fragmentation pattern of complex **3.**

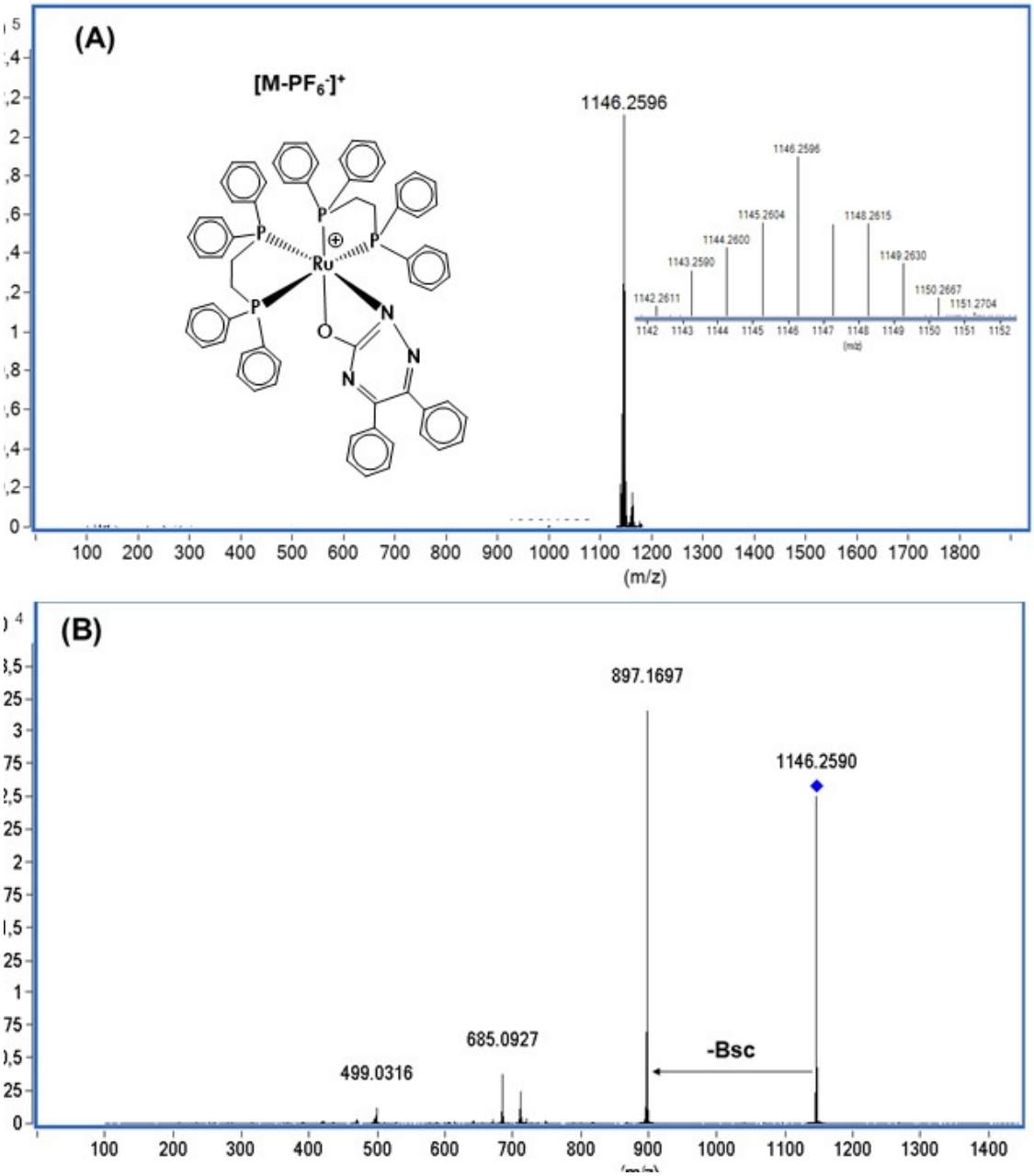

Figure S22. (A) HRESI-MS mass spectrum of complex **4. (B)** HRESI-MS/MS spectrum with fragmentation pattern of complex **4.**