Supplementary Information For:

Novel BODIPY-based Ru (II) and Ir (III) Metalla-rectangles: Cellular Localization of Compounds and Their Antiproliferative Activities

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General Information

The dipyridine-functionalized bodipy ligand[1] and the starting materials (η^6-*p*-cymRu)$_2$(dbq)Cl_2, (η^6-*p*-cymRu)$_2$(dhnq)Cl_2, (η^6-Cp*Ir)$_2$(dbq)Cl_2 and (η^6-Cp*Ir)$_2$(dhnq)Cl_2 were prepared according to reported methods.[2] All other reagents were commercially available and used without further purification. The ^{1}H and ^{13}C{^{1}H}-NMR spectra were recorded on an Agilent 400-MR spectrometer using the residual protonated solvent as internal standard. Infrared spectra were recorded on a Bruker/Hyperion 2000 FTIR spectrometer using KBr pellets. Cyclic voltammetry experiments were carried out in 0.1 M Bu_4NPF_6 solutions using a three-electrode configuration with a WonATech WBCS3000 potentiostat. The compounds were dissolved in dry dichloromethane to give a solution containing 5×10^{-4} M of the rectangles **[1]$^{4+}$-[4]$^{4+}$**. UV-visible absorption spectra were recorded on a Thermo Scientific Genesys 10S UV-Vis spectrometer. Fluorescence spectra were recorded on a Scinco FluoroMate FS-2 fluorescence spectrometer. HRESI-MS were recorded on a Waters SynaptG2S*i* spectrometer at KBSI, South Korea.

Cell culture

Human lung adenocarcinoma (A549), breast adenocarcinoma (MCF-7), cervical adenocarcinoma (HeLa), glioblastoma (U87) and human lung fibroblast (WI-38) cell lines were purchased from the National Centre for Cell Science (NCCS, Pune, India) and maintained in the laboratory. Ham's F-12 medium was used to culture A549 cells whereas MCF-7, HeLa, U87 and WI-38 cells were grown in DMEM. Both media were supplemented with 10 % (v/v) fetal bovine serum (FBS), 100 U/ml Penicillin G, 50 µg/ml Gentamycin sulphate, 100 µg/ml Streptomycin and 2.5 µg/ml Amphotericin B. All cell lines were maintained at 37°C in a humidified atmosphere containing 5% CO_2 in an incubator.

WST-1 cell proliferation assay

For cell culture experiments the stock solutions of the complexes and standard drug cisplatin (Sigma Aldrich, St. Louis, USA) were prepared in DMSO:water in such a manner that the final amount of DMSO exposed to the cell at the highest concentration of treatment remains non-toxic for the cells. Cell proliferation and cell viability were quantified using the WST-1 Cell Proliferation Reagent (Roche diagnostics, Mannheim, Germany), according to the manufacturer's instructions. Principle of this assay is based on cleavage of the water soluble

tetrazolium (WST-1) salt to a formazan by cellular enzymes, especially mitochondrial dehydrogenases (Mosmann 1983). The number of metabolically active cells correlates directly to the amount of formazan generated. All the cell lines were seeded in 96-well culture plates at a density of 1×10^4 cells/well and allowed to settle for 16 h. The cells were then treated with the compounds at increasing doses for 48 h. After treatment, 10 µl of WST-1 cell proliferation reagent was added to each well followed by 3–4 h of incubation at 37°C. Cell proliferation and viability were quantified by measuring the absorbance of the formazan at 460 nm using a microplate ELISA reader MULTISKAN EX (Thermo Electron Corporation, Waltham, MA, USA).

DPPH radical scavenging assay

The antioxidant capacities of the compounds were compared by the DPPH scavenging assay according to literature method with minor modification.[3] Ascorbic acid, a well known antioxidant, was used as a standard compound in the assay. Different concentrations (0-100 µg/ml) of the test and standard compounds were mixed with equal volumes of ethanol. Then 50 µl of DPPH solution (1 mM) was pipetted into the previous mixture and stirred thoroughly. Optical density (OD) of the resulting solution was measured at 517 nm in a Shimadzu UV-2401 PC UV/VIS recording spectrometer (Shimadzu corporation, Kyoto, Japan). All experiments were repeated six times. The percentage radical scavenging activity was calculated from the following formula:

$$\textbf{\% scavenging [DPPH]} = [(A_0 - A_1) / A_0] \times 100$$

Where A_0 was the absorbance of the control (0 µg/ml) and A_1 was the absorbance in the presence of the samples and standard.

Cell cycle analysis

Cell cycle analysis was performed by flow cytometry using the method previously described.[3] All cells were seeded at a density of 1×10^5 cells/well in 12-well plates and incubated at 37°C with 5% CO_2 for 12 h, followed by appropriate treatments of the compounds for 48 h. Cells were harvested and fixed with suitable amounts of chilled methanol and then rehydrated with Phosphate-buffered saline. Cells were then treated with RNase A at 37°C for 1 h to digest cellular RNA. Nuclear DNA was stained with propidium iodide and the cell phase distribution was determined on FACS ARIA (Becton–Dickinson,

New Jersey, USA) equipped with 488 nm Argon (blue) and 633 nm Helium-Neon (red) lasers along with a 623 nm band pass filter using FACS DIVA software (Version 6.0). A histogram of DNA content (x-axis, red fluorescence) versus count (y-axis) was plotted.

Intracellular localization of complexes using confocal microscopy

DAPI (4', 6-diamidino-2-phenylindole) counter staining was done using the method described earlier with certain modifications.[4] All cells were seeded at a density of 2.5×10^5 cells/well in 6-well culture plates containing 22 mm glass coverslips, and allowed to adhere for 16 h. Cells were incubated for 48 h with the compounds at the following concentrations: for MCF-7 cells, 3 μM $[\mathbf{1}]^{4+}$ and 1 μM $[\mathbf{3}]^{4+}$; for HeLa, 3 μM $[\mathbf{1}]^{4+}$; and for U87, 3 μM $[\mathbf{1}]^{4+}$, 3μM $[\mathbf{2}]^{4+}$ and 0.75 μM $[\mathbf{4}]^{4+}$. Post treatment, samples were fixed with 4% paraformaldehyde followed by permeabilization with 0.1% Triton X-100. Cells were stained with 10 μg/ml DAPI for 40 min at room temperature followed by washing to remove unbound dye. The control and treated cells were observed and imaged from ten eye views at 63X magnifications with DAPI and BODIPY specific wavelengths under a Leica TCSSP8 laser scanning confocal microscope (Leica Microsystems, Illinois, USA) and the data was analyzed using Leica Application Suite X software.

DNA binding study

DNA binding behaviors of the complexes were studied by UV-Visible spectroscopy using a Shimadzu UV-2401 PC UV-VIS recording spectrometer (Shimadzu Corporation, Kyoto, Japan). The method described previously by Gupta and co-workers[5] was followed, with certain modifications. Salmon sperm DNA (MP Biomedicals, Ohio, USA) was used for all the experiments. Studies were performed at both 25°C (native) and 95°C (denatured) using 1 cm path length 1 ml quartz cuvettes. Stock solutions of 300 μM complexes in DMSO:water and 200 nM Salmon sperm DNA in KBPES buffer (30 mM Potassium Phosphate with 100 mM KCl, pH 7.0) were prepared. The cuvettes were thoroughly cleaned with distilled water followed by nitric acid (~0.1 N) after each experiment. Two sets, each having 5–25 μM complexes in 400 μl KBPES buffer and 200 nM DNA in 1:1 (v/v) proportions, were prepared. The spectra of the first set having native double stranded DNA (dsDNA) was acquired at 25°C; the other set was acquired after heating the samples at 95°C for 15 minutes so that ds DNA denatures to its single stranded form (ssDNA). All samples were scanned in the range of 200-300 nm. DNA and complex solutions were prepared just before the experiment.

Protein binding study

Solutions of 100 μM Bovine serum albumin (BSA) (MP Biomedicals, Ohio, USA) and 300 μM complexes were prepared in Tris –buffer (pH 7.2). Concentration dependent studies were conducted by mixing increasing amounts of complexes (5–25 μM) with 20 μM BSA in 1:1 (v/v) proportions. Samples were scanned at 25°C (range 200-350 nm) using 1 cm path length 1 ml quartz cuvettes in a Shimadzu UV-2401 PC UV-VIS recording spectrometer (Shimadzu Corporation, Kyoto, Japan).

Statistical analysis

Data from cytotoxicity and DPPH scavenging assays were reported as mean ± SD of six measurements. The statistical analysis was performed using KyPlot version 2.0 beta 15 (32 bit). The IC_{50} values were calculated by the formula, $Y = 100 \times A_1/(X + A_1)$ where $A_1 = IC_{50}$, Y = response (Y = 100 % when X = 0), X = inhibitory concentration.

Synthetic procedures for rectangles $[1]^{4+}$-$[4]^{4+}$

Synthesis of $[\mathbf{1}]^{4+}$: A mixture of $(\eta^6\text{-p-cymRu})_2Ru_2(\mu_4\text{-dbq})Cl_2$ (43 mg, 0.06 mmol) and $AgCF_3SO_3$ (33 mg, 0.13 mmol) in methanol (25 mL) was stirred at room temperature for 4 h and then filtered to remove AgCl. To the dark green filtrate, bodipy ligand (30 mg, 0.06 mmol) was added and the solution was refluxed for 15 h, then the solvent was removed under vacuum. The residue was dissolved in dichloromethane (3 mL), and hexane was slowly added to precipitate the dark solid. The solid was removed by filtration and dried under vacuum.

Yield 75 mg (85%). ESI MS (CH_3CN): *m/z* = 544.84 $[M - 4CF_3SO_3]^{4+}$. IR (KBr pellets): ν = 3489 (b, C-H_{ar}), 1616 (s, C=N), 1523 (s, C=O), 1255 (s, CF_3). ^1H NMR (400 MHz, CD_2Cl_2): δ = 8.27 (d, 8H, *J* = 8, H_α), 7.62 (m, 6H, H_{Ph}), 7.42 (d, 4H, *J* = 8, H_{Ph}), 7.23 (d, 8H, *J* = 8, H_β), 5.85 (d, 8H, *J* = 4, $H_{p\text{-cym}}$), 5.84 (s, 4H, H_{dbq}), 5.71 (d, 8H, *J* = 8, $H_{p\text{-cym}}$), 2.84 (sept, 4H, *J* = 8, C**H**$(CH_3)_2$), 2.40 (s, 12H, CH_3), 2.18 (s, 12H, C**H**$_3C_{cym}$), 1.38 (s, 12H, CH_3), 1.34 (d, 24H, *J* = 8, CH(C**H**$_3)_2$). $^{13}C\{^1H\}$-NMR (100 MHz, CD_2Cl_2): δ = 184.37, 165.46, 152.08, 132.22, 126.93, 112.83, 109.99, 31.43, 29.60, 21.98, 12.55.

Synthesis of [**2**]$^{4+}$: A mixture of $(\eta^6$-p-cymRu$)_2$Ru$_2(\mu_4$-dhnq$)$Cl$_2$ (46 mg, 0.06 mmol) and $AgCF_3SO_3$ (33 mg, 0.13 mmol) in methanol (25 mL) was stirred at room temperature for 4 h and then filtered to remove AgCl. To the dark green filtrate, bodipy ligand (30 mg, 0.06 mmol) was added and the solution was refluxed for 15 h, then the solvent was removed under vacuum. The residue was dissolved in dichloromethane (3 mL), and hexane was slowly added to precipitate the dark solid. The solid was removed by filtration and dried under vacuum.

Yield 91 mg (77 %). ESI MS (CH_3CN): *m/z* = 572.88 [M - 4CF_3SO_3]$^{4+}$. IR (KBr pellets): ν = 3504 (b, C-H_{ar}), 1616 (s, C=N), 1539 (s, C=O), 1272 (s, CF_3). ^{1}H NMR (400 MHz, CD_2Cl_2): δ = 8.39 (d, 8H, *J* = 4, H_α), 7.62 (m, 6H, H_{Ph}), 7.38 (d, 4H, *J* = 8, H_{Ph}), 7.23 (s, 8H, H_{dhnq}), 7.19 (d, 8H, *J* = 8, H_β), 5.66 (d, 8H, *J* = 4, $H_{p\text{-cym}}$), 5.50 (d, 8H, *J* = 4, $H_{p\text{-cym}}$), 2.78 (sept, 4H, *J* = 8, C**H**$(CH_3)_2$), 2.37 (s, 12H, CH_3), 2.08 (s, 12H, C**H**$_3C_{cym}$), 1.35 (s, 12H, CH_3), 1.31 (d, 24H, *J* = 8, CH(C**H**$_3)_2$). ^{13}C{^{1}H}-NMR (100 MHz, CD_2Cl_2): δ = 171.03, 154.22, 151.41, 145.11, 141.16, 137.36, 134.09, 129.74, 127.54, 126.95, 111.75, 110.06, 103.68, 99.36, 30.68, 21.89, 17.04, 13.08, 12.49.

Synthesis of [**3**]$^{4+}$: A mixture of $(\eta^6$-Cp*Ir$)_2(\mu_4$-dbq$)$Cl$_2$ (54 mg, 0.06 mmol) and $AgCF_3SO_3$ (33 mg, 0.12 mmol) in methanol (25 mL) was stirred at room temperature for 4 h and then filtered to remove AgCl. To the dark brown filtrate, bodipy ligand (30 mg, 0.04 mmol) was added and the solution was refluxed for 15 h, then the solvent was removed under vacuum. The residue was dissolved in dichloromethane (3 mL), and hexane was slowly added to precipitate the dark solid. The solid was removed by filtration and dried under vacuum.

Yield 90 mg (92%). ESI MS (CH_3CN): *m/z* = 637.30 [M - 4CF_3SO_3]$^{4+}$. IR (KBr pellets): ν = 3498 (b, C-H_{ar}), 1616 (s, C=N), 1531 (s, C=O), 1261 (s, CF_3). ^{1}H NMR (400 MHz, CD_2Cl_2): δ = 8.31 (d, 8H, *J* = 8, H_α), 7.61 (m, 6H, H_{Ph}), 7.43 (d, 4H, *J* = 8, H_{Ph}), 7.37 (d, 8H, *J* = 8, H_β), 5.99 (s, 4H, H_{dbq}), 2.48 (s, 12H, CH_3), 1.60 (s, 60H, Cp*), 1.40 (s, 12H, CH_3). ^{13}C{^{1}H}-NMR (100 MHz, CD_2Cl_2): δ = 185.44, 154.18, 151.29, 145.97, 141.21, 133.94, 131.80, 129.77, 128.09, 127.57, 109.99, 101.95, 13.13, 12.68, 8.58.

Synthesis of $[\mathbf{4}]^{4+}$: A mixture of $(\eta^6\text{-Cp*Ir})_2(\mu_4\text{-dhnq})Cl_2$ (57 mg, 0.06 mmol) and $AgCF_3SO_3$ (33 mg, 0.13 mmol) in methanol (25 mL) was stirred at room temperature for 4 h and then filtered to remove AgCl. To the dark brown filtrate, bodipy ligand (30 mg, 0.06 mmol) was added and the solution was refluxed for 15 h, then the solvent was removed under vacuum. The residue was dissolved in dichloromethane (3 mL), and hexane was slowly added to precipitate the dark solid. The solid was removed by filtration and dried under vacuum.

Yield 90 mg (89%). ESI MS (CH_3CN): *m/z* = 661.19 $[M - 4CF_3SO_3]^{4+}$. IR (KBr pellets): ν = 3506 (b, C-H_{ar}), 1616 (s, C=N), 1537 (s, C=O), 1269 (s, CF_3). ^{1}H NMR (400 MHz, CD_2Cl_2): δ = 8.39 (d, 8H, *J* = 4, H_α), 7.61 (m, 6H, H_{Ph}), 7.40 (d, 4H, *J* = 8, H_{Ph}), 7.36 (s, 8H, H_{dhnq}), 7.32 (d, 8H, *J* = 8, H_β), 2.43 (s, 12H, CH_3), 1.48 (s, 60H, Cp*), 1.37 (s, 12H, CH_3). $^{13}C\{^1H\}$-NMR (100 MHz, CD_2Cl_2): δ = 168.18, 154.17, 149.69, 145.83, 140.95, 138.79, 133.91, 129.77, 127.82, 127.53, 113.64, 110.00, 13.12, 12.56, 8.08.

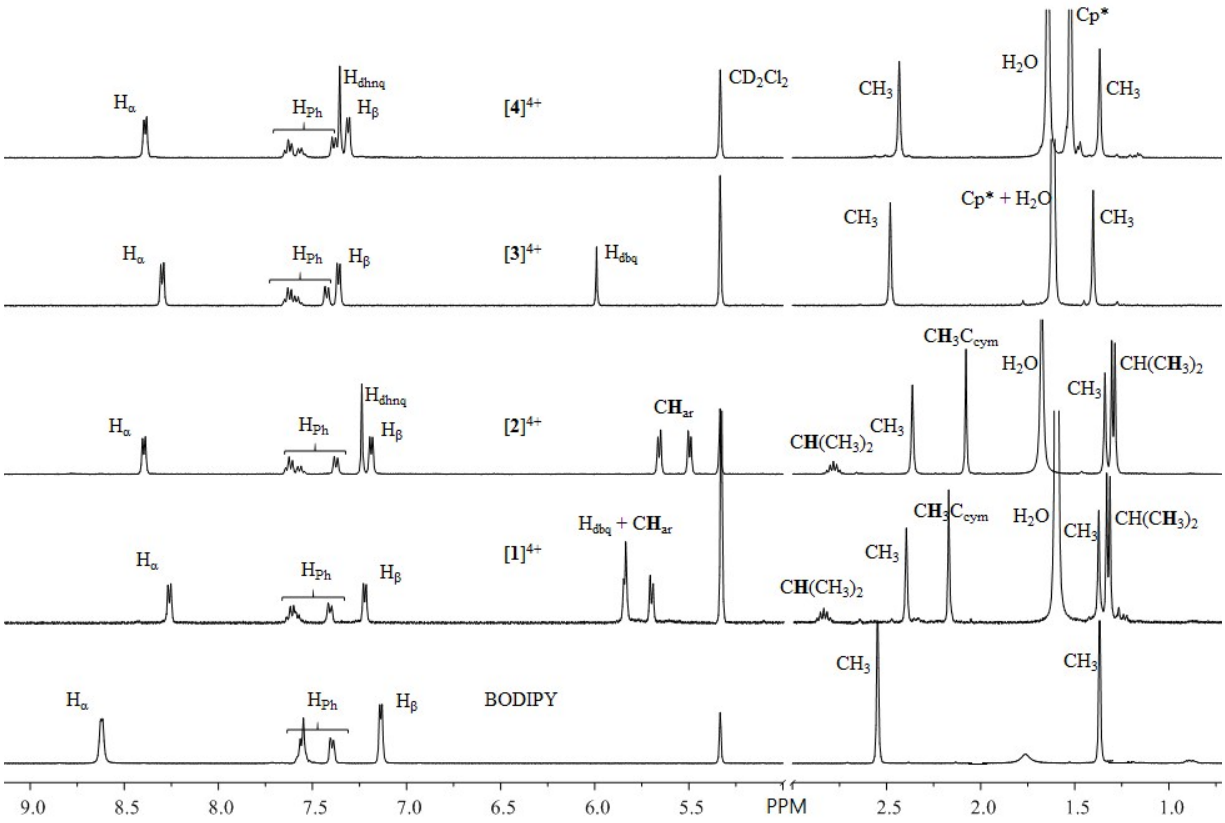

Fig. S1. ^{1}H NMR spectra of complexes $[\mathbf{1}]^{4+}$-$[\mathbf{4}]^{4+}$ and free BODIPY in CD_2Cl_2.

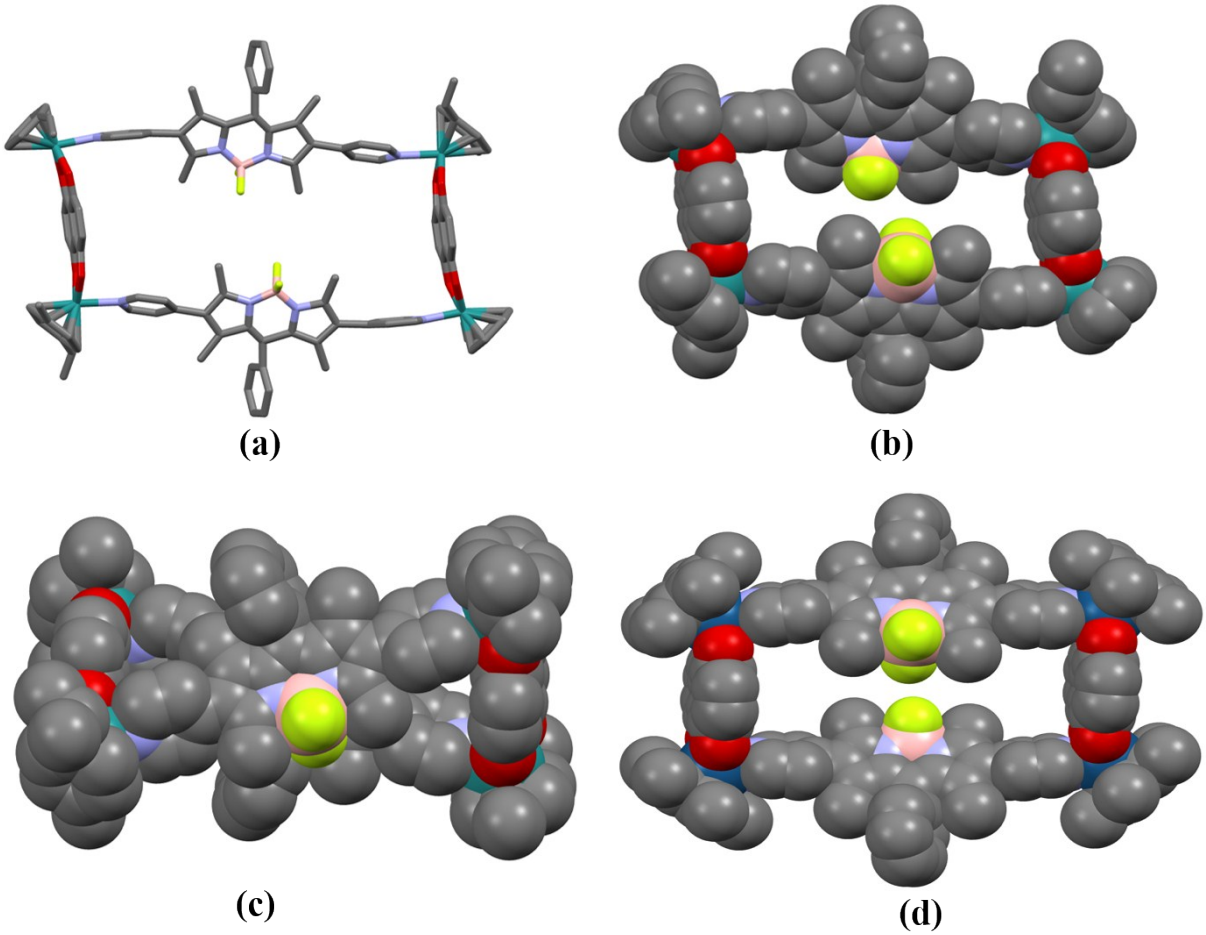

Fig. S2. X-ray structure of planar [**2**]$^{4+}$ (a) and space-filling model of planar [**2**]$^{4+}$ (b), twisted [**2**]$^{4+}$ (c) and [**4**]$^{4+}$ (d). Counter anions, hydrogen atoms and solvent molecules are omitted for clarity.

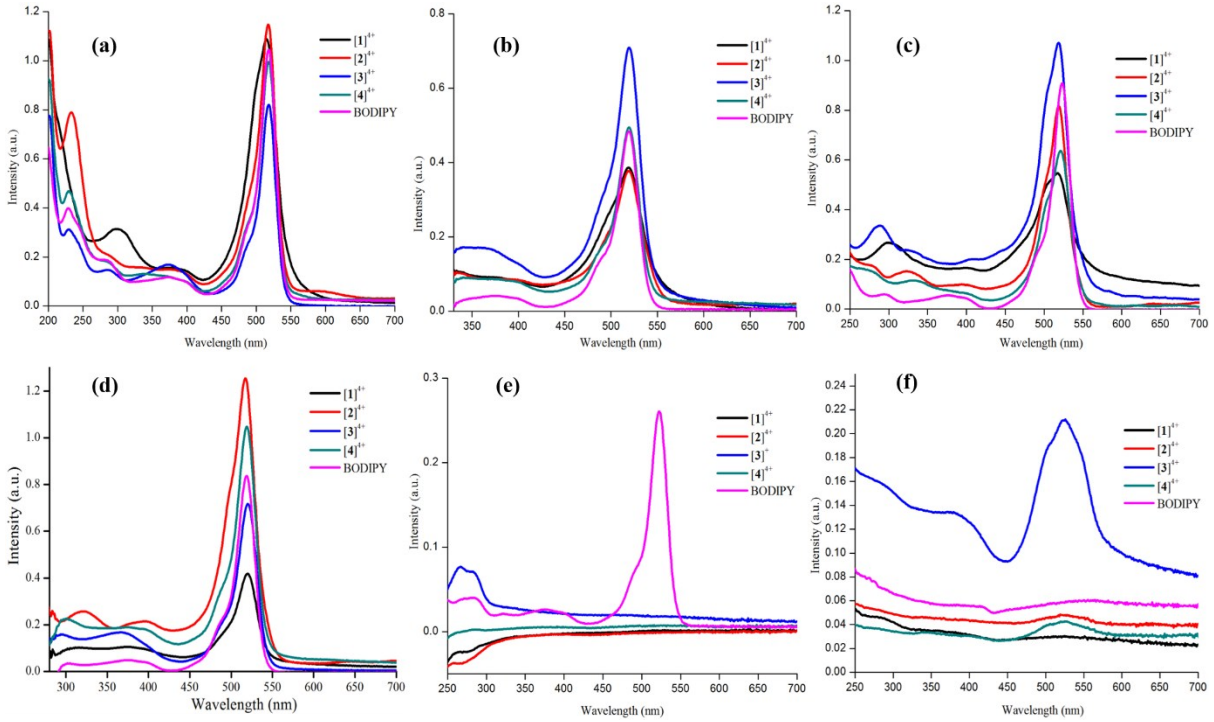

Fig. S3. UV-vis absorption spectra of rectangles $[\mathbf{1}]^{4+}$- $[\mathbf{4}]^{4+}$ and BODIPY in (a) acetonitrile, (b) acetone, (c) chloroform, (d) ethanol, (e) hexane and (f) water in (1×10^{-5} M) solution.

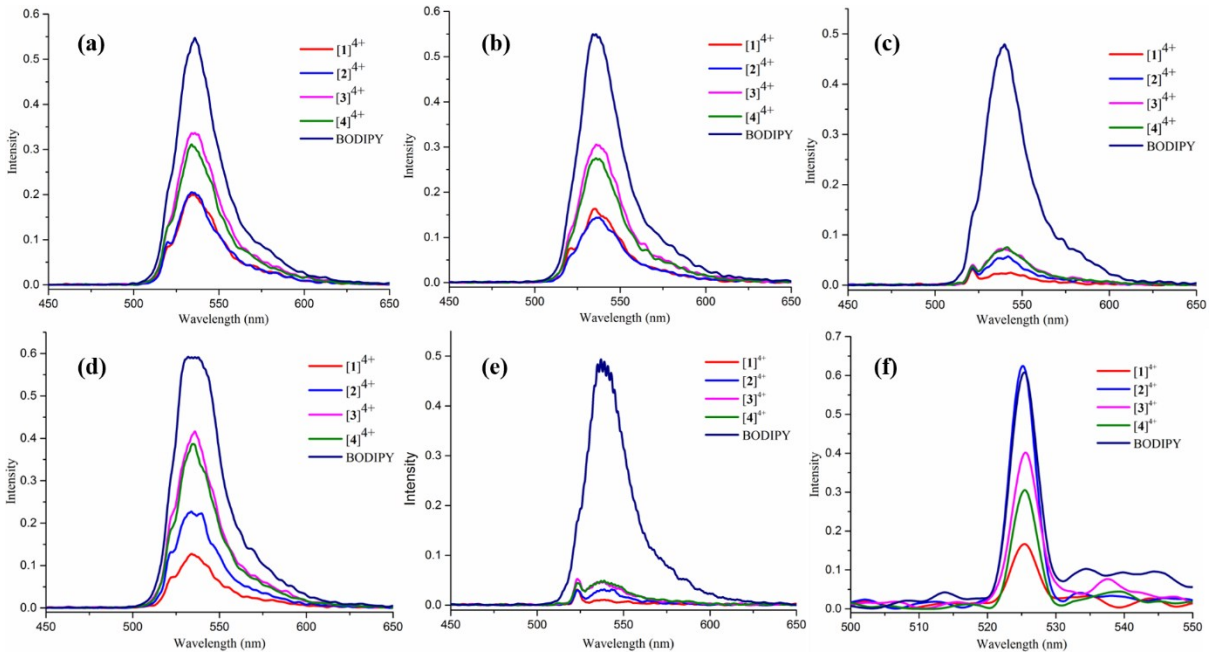

Fig. S4. Photoluminescent spectra of rectangles $[\mathbf{1}]^{4+}$- $[\mathbf{4}]^{4+}$ and BODIPY in (a) acetonitrile, (b) acetone, (c) chloroform, (d) ethanol, (e) hexane and (f) water in (1×10^{-7} M) solution.

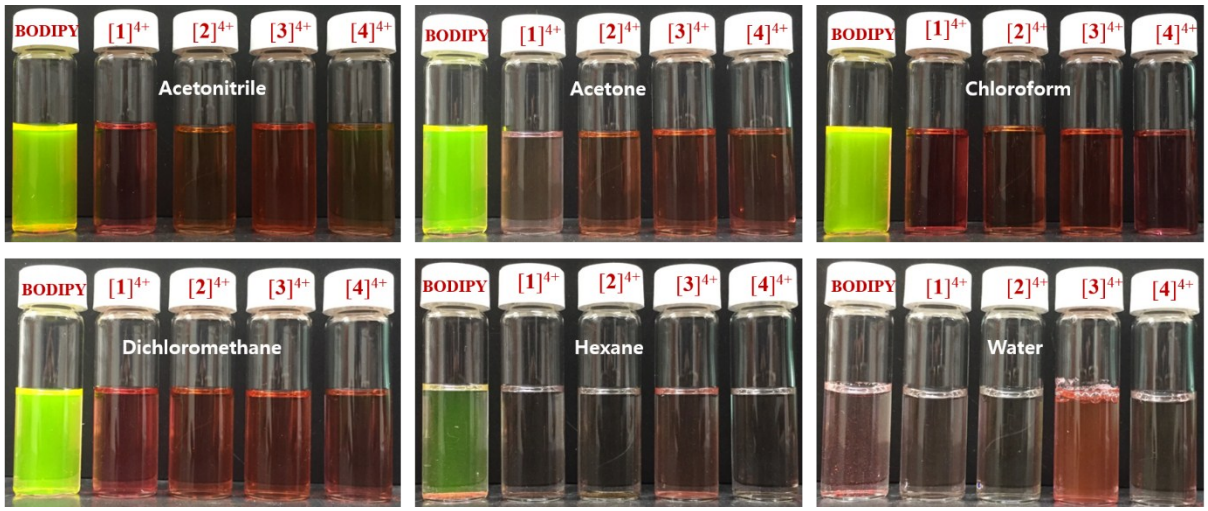

Fig. S5. Photographs of BODIPY and complexes [**1**]$^{4+}$- [**4**]$^{4+}$ in different solvents in white light in (1×10^{-5} M) solution.

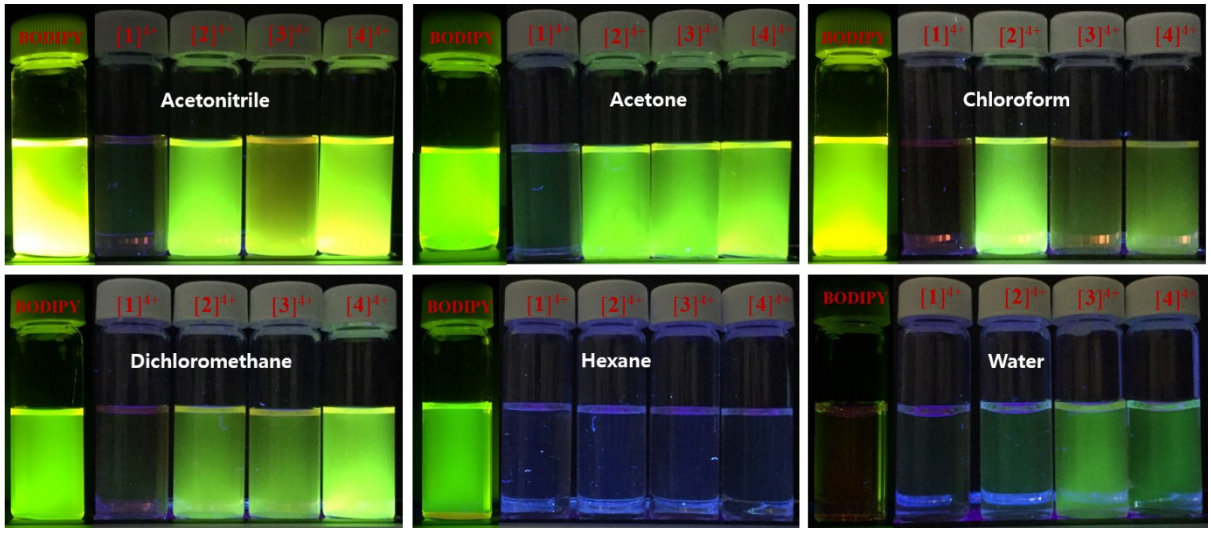

Fig. S6. Photographs of BODIPY and complexes [**1**]$^{4+}$- [**4**]$^{4+}$ in different solvents in UV light in (1×10^{-5} M) solution.

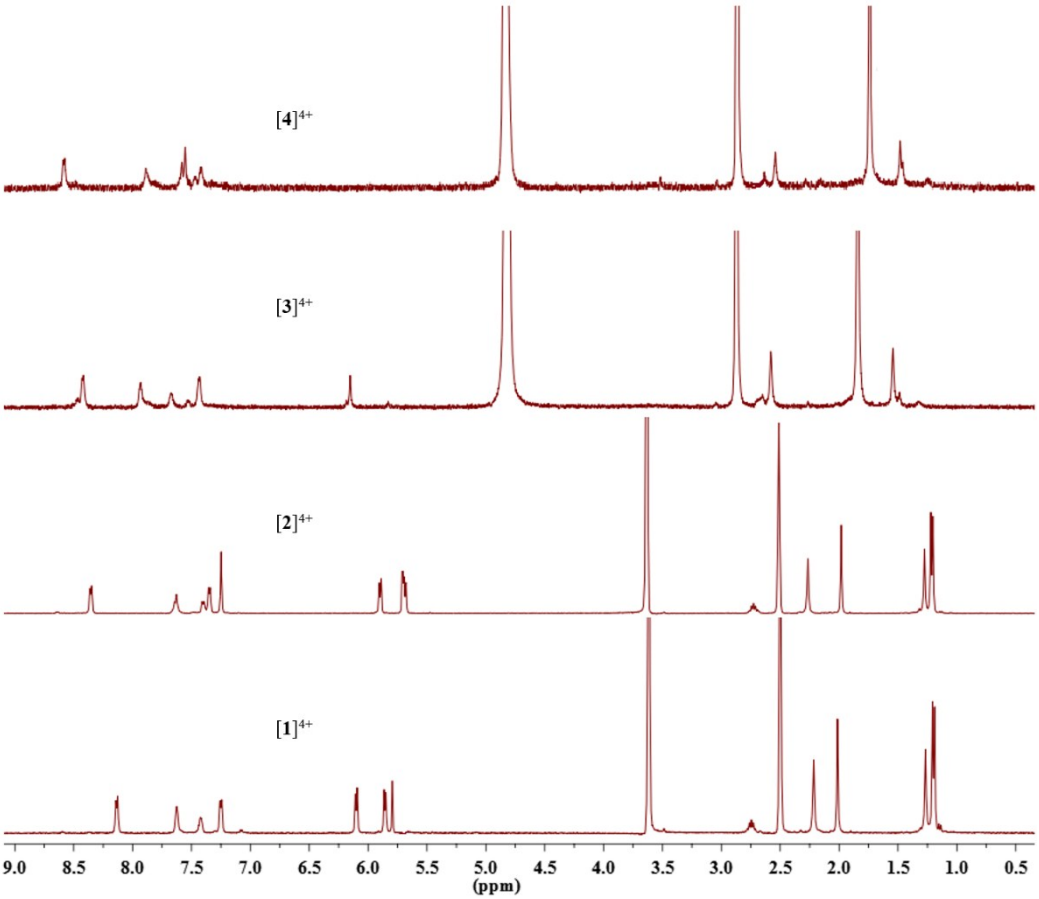

Fig. S7. ^{1}H NMR spectra of complexes [**1**]$^{4+}$-[**4**]$^{4+}$in DMSO-d_6/D_2O after 24h. For complex [**1**]$^{4+}$ and [**2**]$^{4+}$, the ratio of DMSO-d_6/D_2O used is 1:1 and for complexes [**3**]$^{4+}$ and [**4**]$^{4+}$ it is 1:4 ratio.

Cyclic Voltammetry:

The presence of the redox-active metal centers makes the rectangles $[\mathbf{1}]^{4+}$-$[\mathbf{4}]^{4+}$ ideal candidates for analysing their redox properties. All complexes show a rev-ersible oxidation step between -0.89 to 1.34 V (figure S8) in CH_2Cl_2/0.1 M Bu_4NPF_6. This result indicates that the shift in metal center from Ru(II) to Ir(III) and ligand from benzoquinone to naphthoquinone have less effect on its potent-ials.

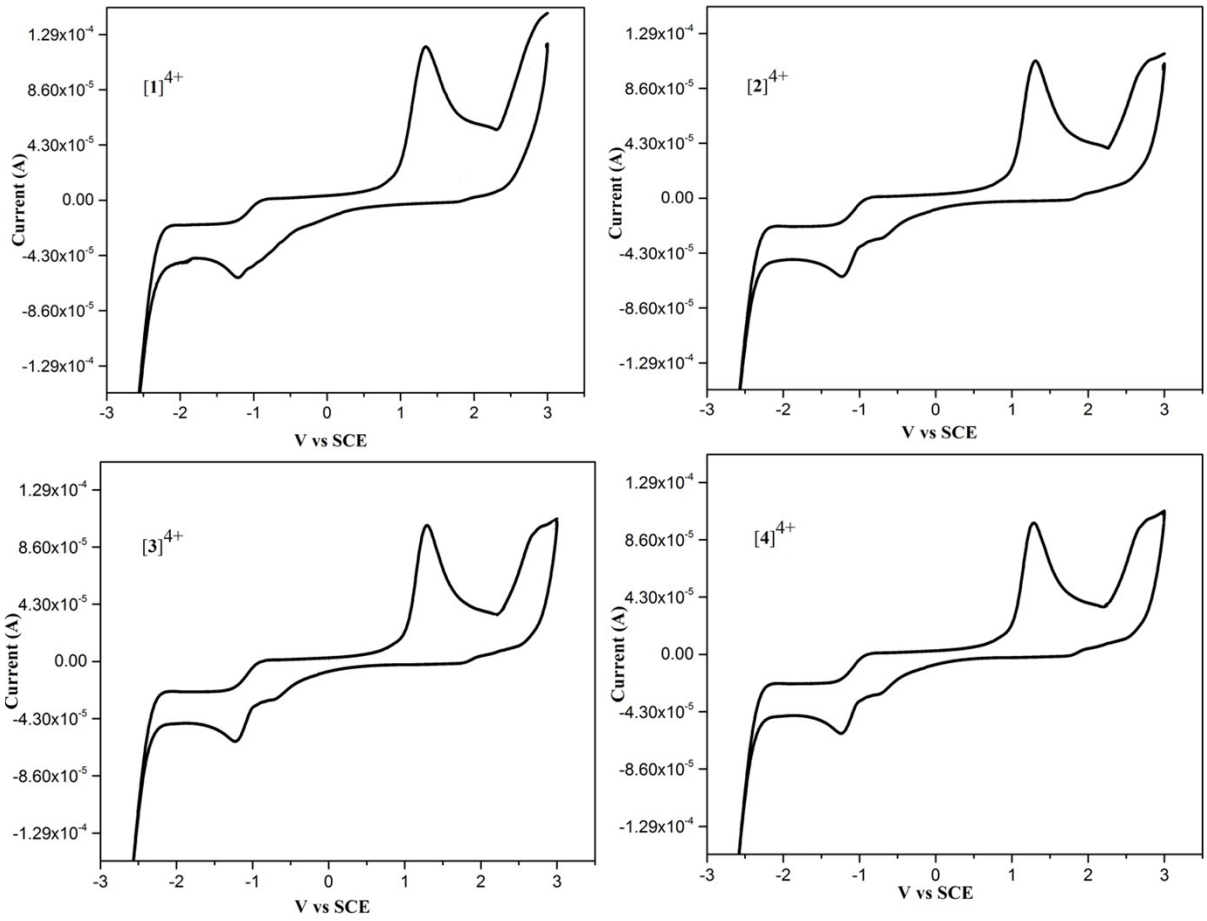

Fig. S8. Cyclic voltammograms of 0.5 mM $[\mathbf{1}]^{4+}$ - $[\mathbf{4}]^{4+}$ in CH_2Cl_2/0.1 M Bu_4NPF_6 at 298 K.

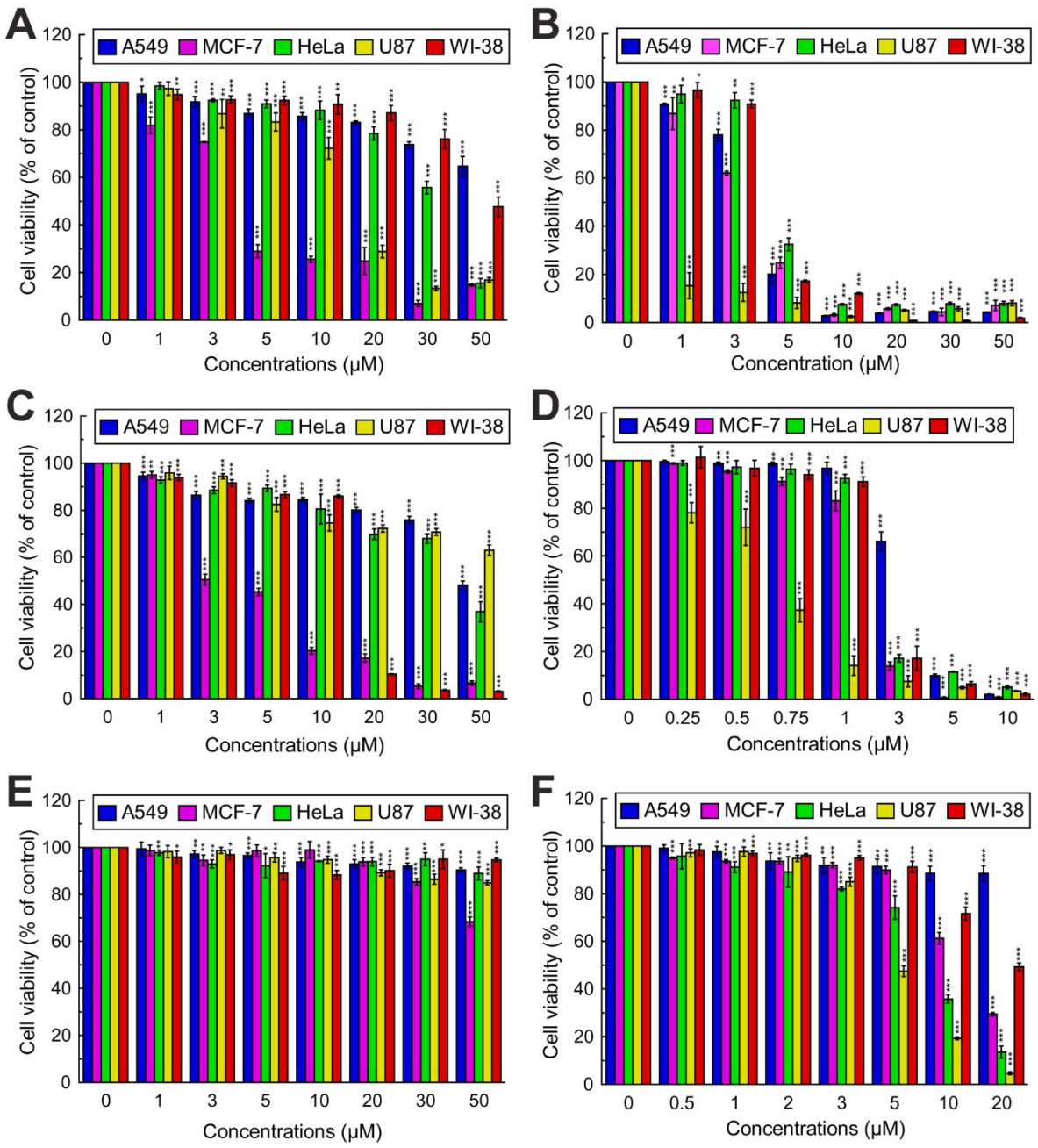

Fig. S9. Effect of (A) $[\mathbf{1}]^{4+}$, (B) $[\mathbf{2}]^{4+}$, (C) $[\mathbf{3}]^{4+}$, (D) $[\mathbf{4}]^{4+}$, (E) BODIPY, and (F) Cisplatin on the cell proliferation and viability of cancer (A549, MCF-7, HeLa & U87) and normal (WI-38) cells. Cells were treated with increasing concentrations of the compound for 48 h; cell proliferation and viability was determined with WST-1 cell proliferation reagent. Results were expressed as cell viability (% of control).

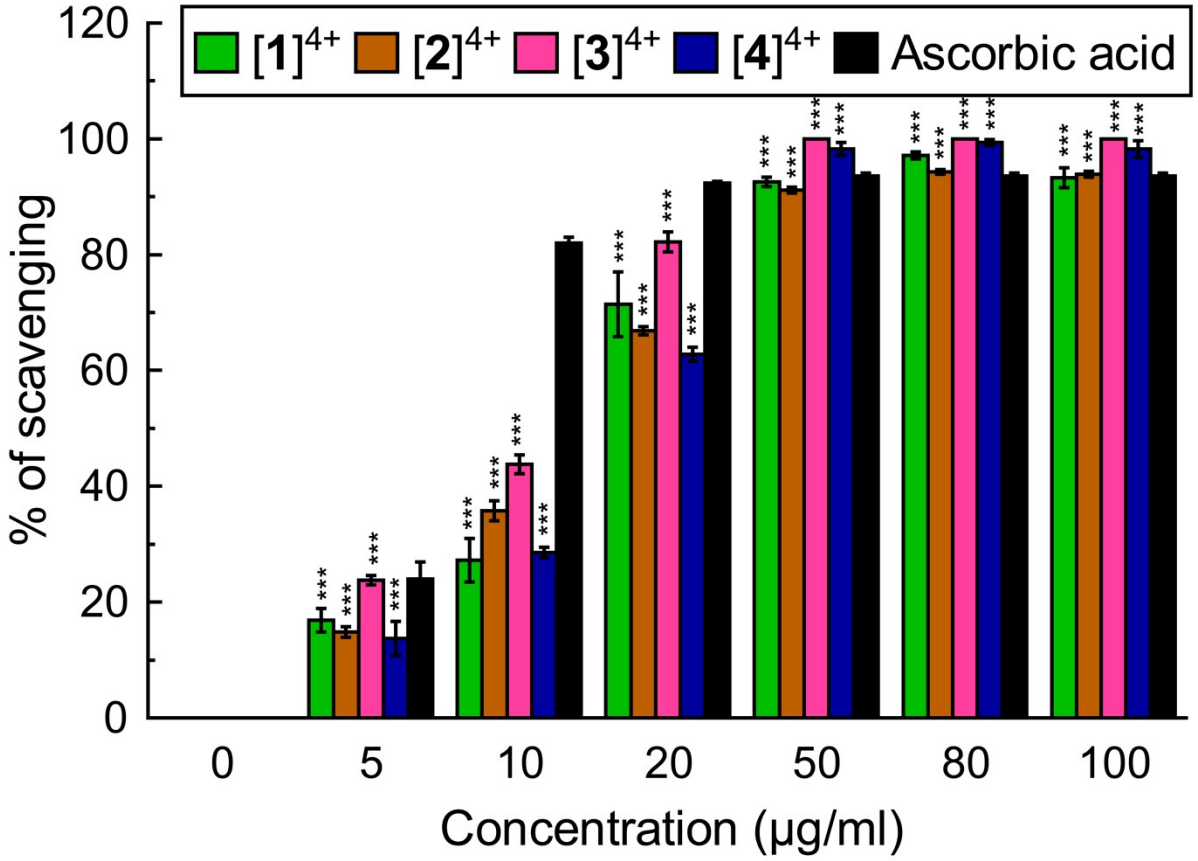

Fig. S10. DPPH radical scavenging activity of $[\mathbf{1}]^{4+}$- $[\mathbf{4}]^{4+}$ compared with ascorbic acid, a standard antioxidant reference compound. All data are expressed as mean ± SD (n=6). *P < 0.05, **P < 0.01 and ***P < 0.001 vs. 0 μg/ml.

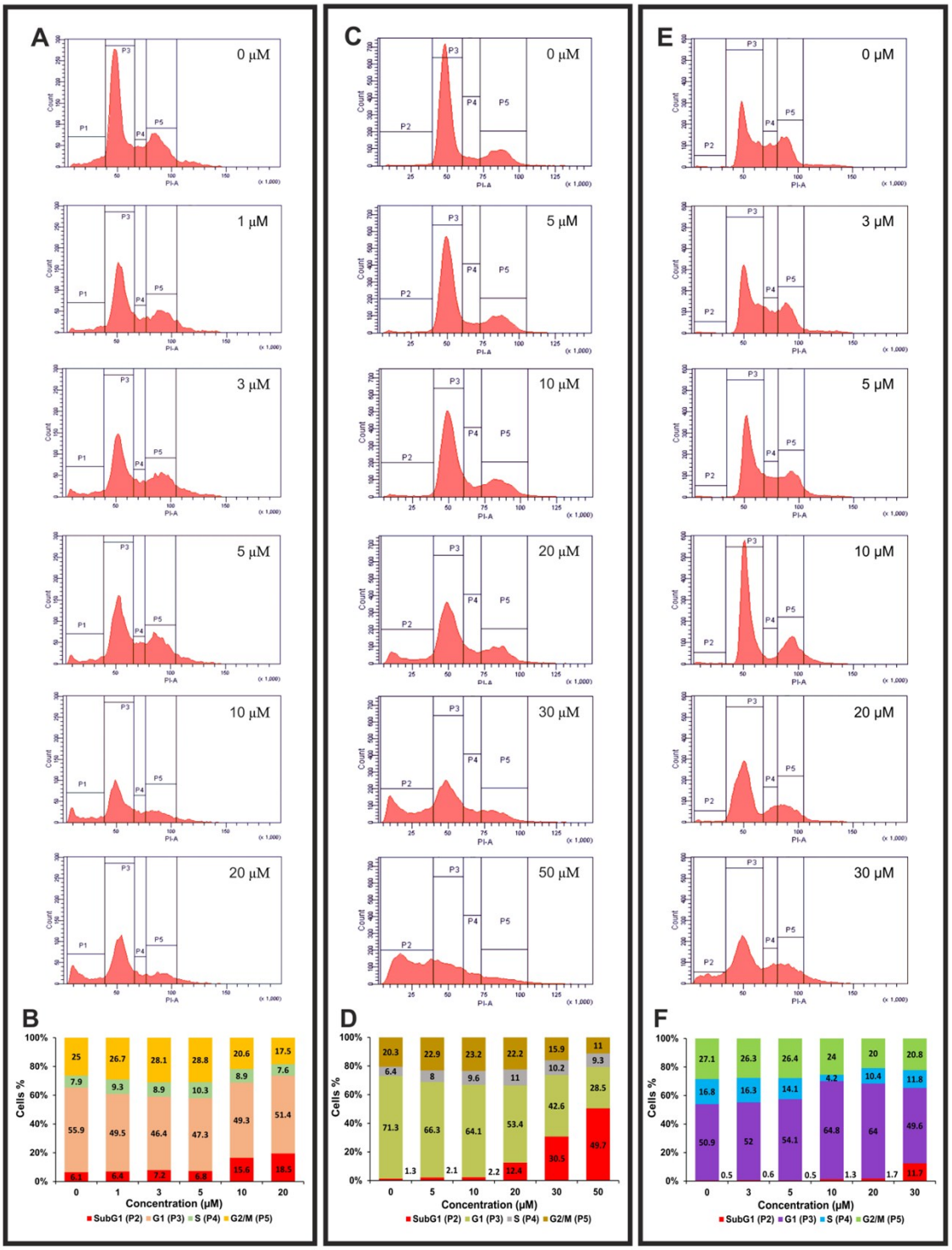

Fig. S11. Cell cycle analysis of cells treated with $[\mathbf{1}]^{4+}$ using flow cytometry. Sub-G1, G1, S and G2/M phases of (A) MCF-7, (C) HeLa and [E] U87 cells, treated with increasing doses of $[\mathbf{1}]^{4+}$ for 48 hours. Graphical representation of % cell population in different phases for MCF-7 (B), HeLa (D) and U87 (F) cells. The respective percentages of cells present in each cell cycle phase are mentioned on the bars.

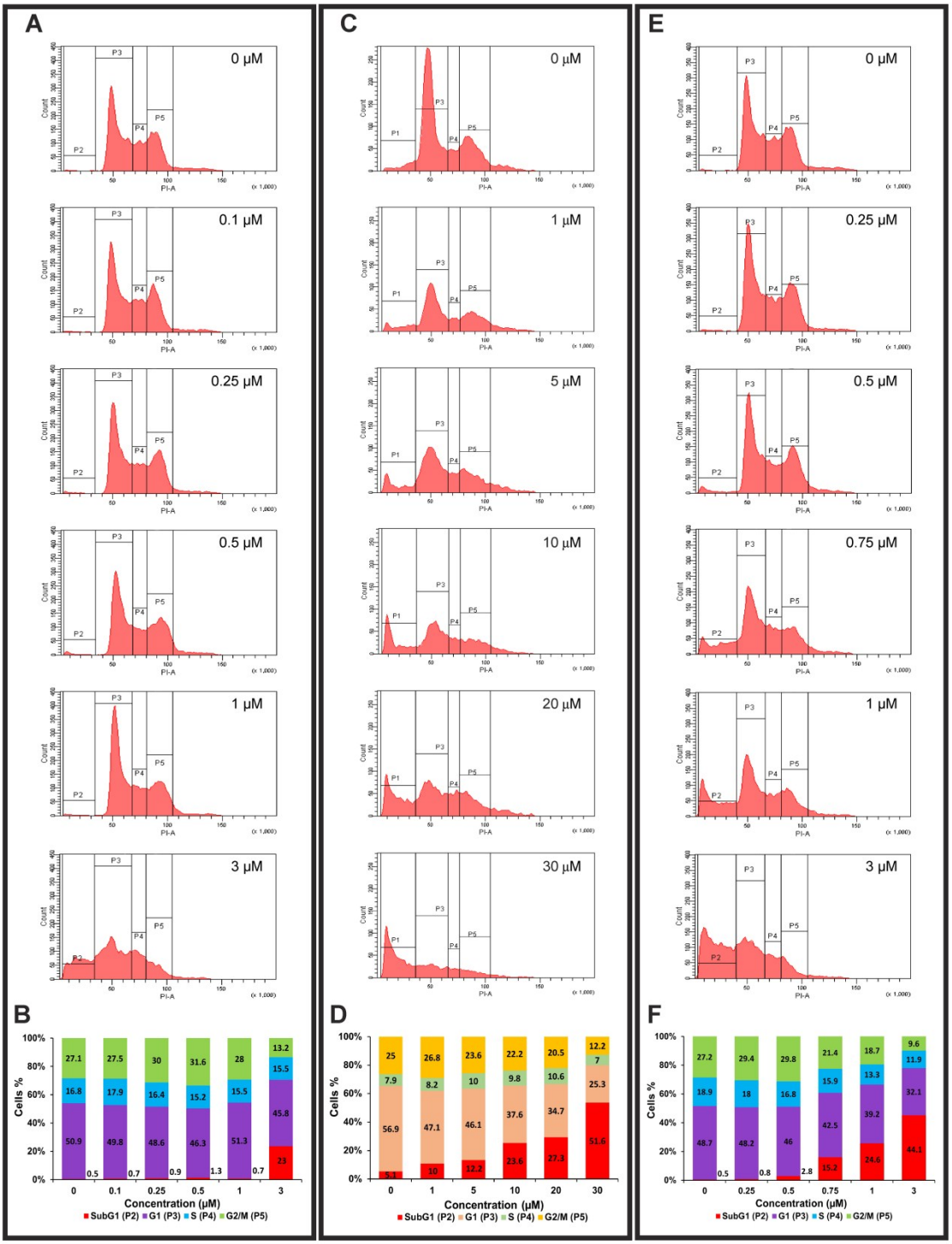

Fig. S12**.** Cell cycle analysis of cells treated with $[\mathbf{2}]^{4+}$, $[\mathbf{3}]^{4+}$ and $[\mathbf{4}]^{4+}$ using flow cytometry. Sub-G1, G1, S and G2/M phases of (A) U87 cells treated with increasing doses of $[\mathbf{2}]^{4+}$, (C) MCF-7 with $[\mathbf{3}]^{4+}$, and (E) U87 cells with $[\mathbf{4}]^{4+}$, for 48 hours. (B), (D) and (F) illustrate the % cell population in different phases, graphically for (A), (C) and (E), respectively. The respective percentages of cells present in each cell cycle phase are mentioned on the bars.

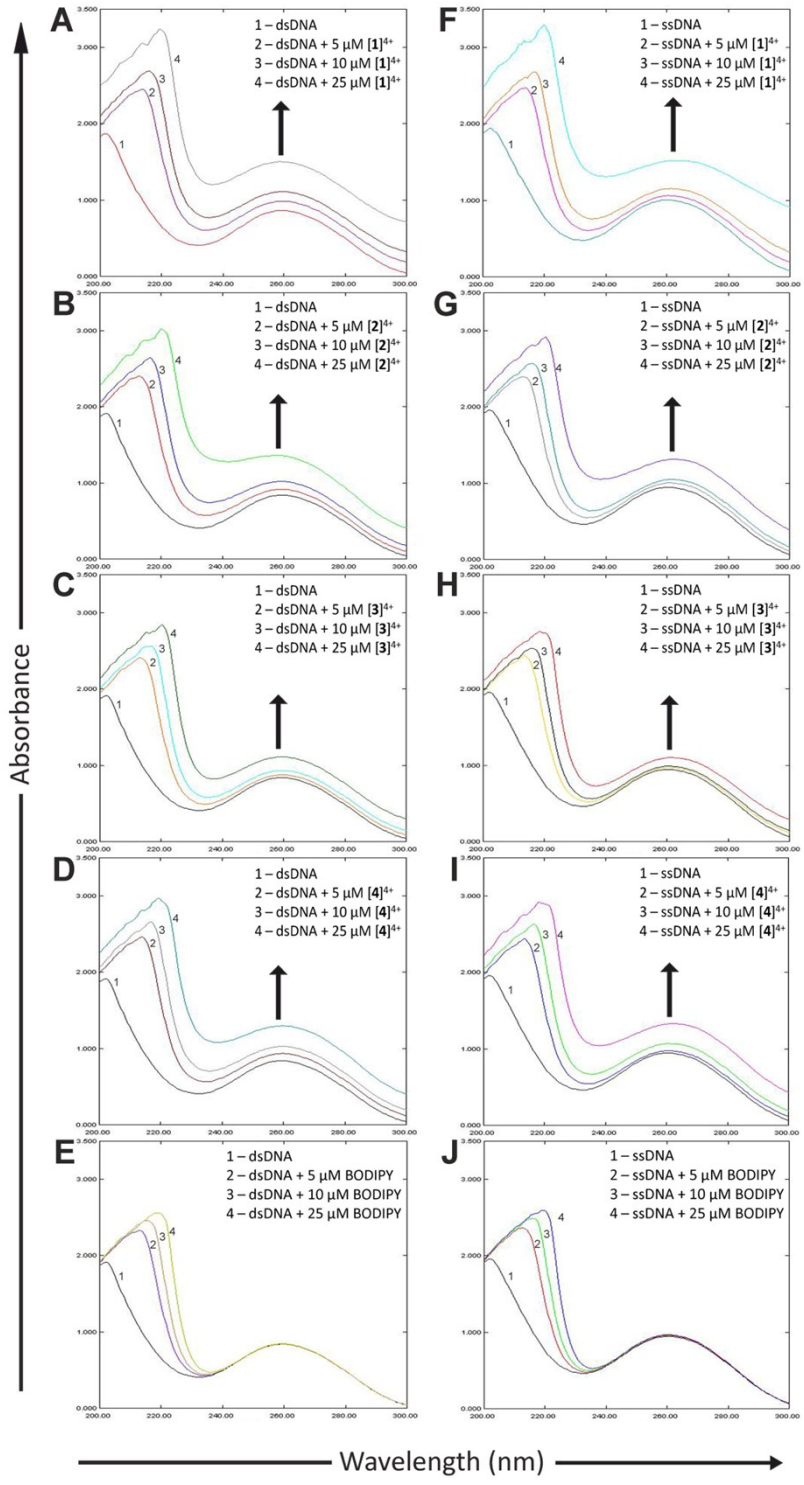

Fig. S13. UV visible spectra obtained with increasing concentrations of metalla-rectangles [**1**]$^{4+}$- [**4**]$^{4+}$ and BODIPY ligand upon interaction with Salmon sperm DNA. (A-D) dsDNA + complexes and (E) dsDNA + BODIPY ligand under native conditions (25°C). (F-I) ssDNA + complexes and (J) ssDNA + BODIPY ligand under denaturing conditions (95°C). Conditions of each spectra are marked with appropriate numbering.

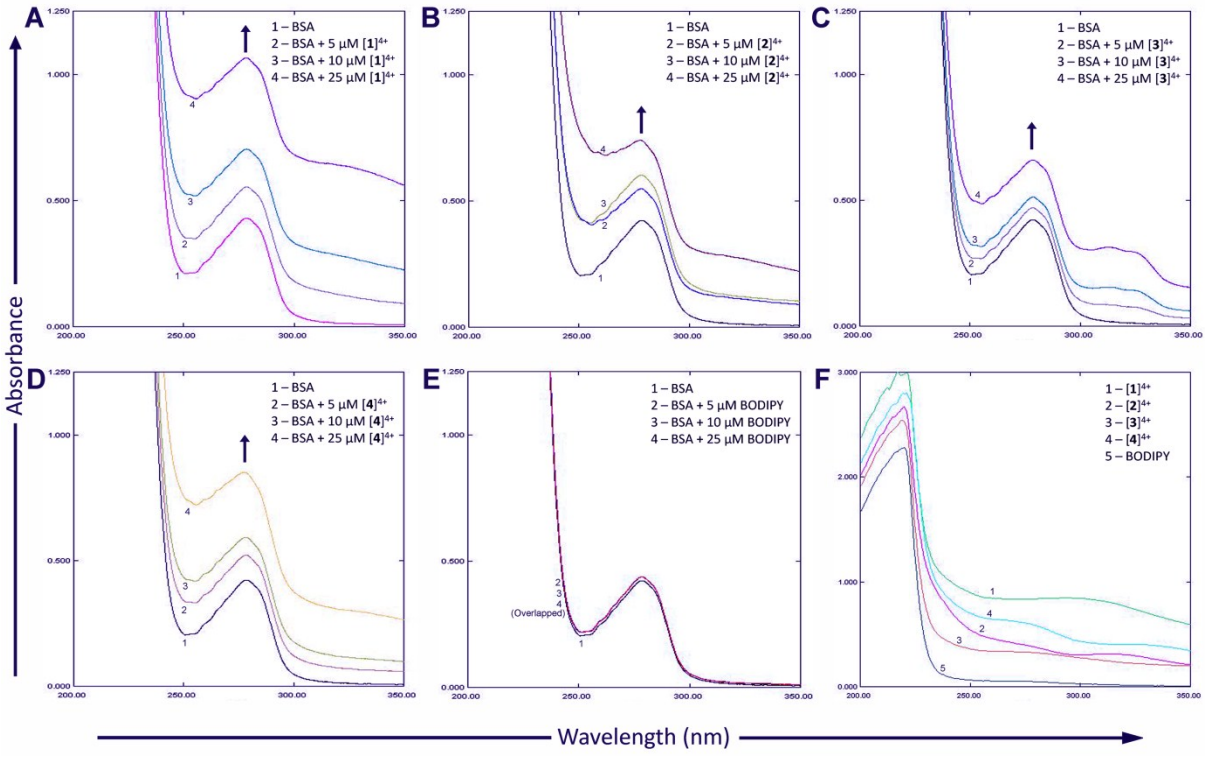

Fig. S14. UV visible spectra obtained with increasing concentrations of metalla-rectangles [**1**]$^{4+}$- [**4**]$^{4+}$ and BODIPY ligand upon interaction with BSA protein at 25°C. (A-D) BSA + complexes. (E) BSA + BODIPY ligand. (F) Spectra of individual complexes without BSA. Conditions of each spectra are marked with appropriate numbering.

Single-crystal X-ray structure analyses

Reflection data for [**2**]$^{4+}$ and [**4**]$^{4+}$ were collected using a Bruker APEX-II CCD-based diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.7107 Å). Hemispheres of reflection data were collected as ω scan frames with 0.5°/frame and an exposure time of 5 s/frame. Cell parameters were determined and refined using the SMART program. Data reduction was performed using SAINT software. The data were corrected for Lorentz and polarization effects. An empirical absorption correction was applied using the SADABS program. The structures of the compounds were solved using direct methods and refined by full-matrix least-squares methods using the SHELXTL program package with anisotropic thermal parameters for all non-hydrogen atoms. The crystals of [**2**]$^{4+}$ and [**4**]$^{4+}$ diffracted very weakly because of the large amounts of disordered solvents and anions. Geometrical restraints, i.e., DFIX, SADI, SIMU, and AFIX 66, on part of the hexagonal aromatic rings were used in the refinements. Because of complicated disorder, one of triflate ions in [**4**]$^{4+}$

was removed by SQUEEZE method of PLATON. Crystallographic details for [**2**]$^{4+}$ and [**4**]$^{4+}$ are summarized in Table S1. Figures of molecular structures were drawn with MERCURY program. CCDC 1430114 for twisted [**2**]$^{4+}$, 1430115 for planar [**2**]$^{4+}$ and 1430116 for [**4**]$^{4+}$ contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) +44-1223/336-033; Email: deposit@ccdc.cam.ac.uk].

Note: Some A- and B-level alerts were found using the IUCR's CheckCIF routine for complexes [**2**]$^{4+}$ and [**4**]$^{4+}$, all of which originated from the limited diffraction ability of this type of supramolecular compound in the crystal state.

Table S1. Crystallographic data and parameters for [**2**]$^{4+}$ and [**4**]$^{4+}$

	twisted [**2**]$^{4+}$	[**2**]$^{4+}$	[**4**]$^{4+}$
formula	$C_{162}H_{104}B_2F_{16}N_8O_{20}Ru_4S_4$	$C_{126.6}B_2F_8N_8O_{12.6}Ru_4S_2$	$C_{134}H_{150}B_2F_{10}Ir_4N_8O_{16}S_2$
formula weight	2860.26	2478.03	3205.16
crystal system	Monoclinic	Triclinic	Monoclinic
space group	*C2/c*	*P-1*	$P2_1/c$
a (Å)	24.5419(6)	24.488(2)	20.4184(9)
b (Å)	22.5400(6)	24.507(3)	19.7746(9)
c (Å)	25.4485(7)	25.043(4)	20.8298(9)
α (°)	90	60.821(9)	90
β (°)	111.767(2)	61.007(9)	113.735(2)
γ (°)	90	60.124(5)	90
V (Å^3)	13073.7(6)	10782(3)	7699.0(6)
Z	4	3	2
ρ_{calc} (g cm^{-3})	1.453	1.145	1.383
μ (mm^{-1})	0.606	0.503	3.543
F(000)	5784	3622	3188
T (K)	100(2)	100(2)	100(2)
scan mode	ω and ϕ	ω and ϕ	ω and ϕ
hkl range	-29 ≤ h ≤ 29,	-16 ≤ h ≤ 16,	-23 ≤ h ≤ 22,
	-27 ≤ k ≤24,	-16 ≤ k ≤ 16,	-20 ≤ k ≤ 18,
	-29 ≤ l ≤ 30	-16 ≤ l ≤ 16	-19 ≤ l ≤ 23
measd reflns	84240	36670	39983
unique reflns [R_{int}]	12014[0.0452]	6914	10982[0.0568]
refined parameters	823	761	820
R_1[a] (I > 2σ(I))	0.0746	0.2233	0.0851
wR_2[b] all data	0.1967	0.4644	0.2121
GOF on F^2	1.049	2.513	1.054
ρ_{fin} (max/min) (e Å^{-3})	2.031 / -1.289	1.607 / -1.008	4.502 / -3.680

[a] $R1 = \sum||Fo| - |Fc||/\sum|Fo|$. [b] $wR2 = \{[\sum w(Fo^2 - Fc^2)^2]/[\sum w(Fo^2)^2]\}^{1/2}$.

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