

Supporting Information for:

A gold(I) complex with a photoactive ligand behaves as two-
in-one dual metallaphotoredox cross-coupling catalyst

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1.- General considerations.

Imidazolium salt **A** and complex **1** were prepared as previously reported by us.^[1] The aryldiazonium tetrafluoroborate compounds were prepared according to literature methods.^[2-3] All the other reagents were used as received from the commercial suppliers. Anhydrous solvents were dried using a solvent purification system (SPS M BRAUN) or purchased and degassed prior to use by purging them with dry nitrogen. NMR spectra were recorded on a Bruker 300 MHz, using CDCl₃ or CD₃CN as solvents. ESI mass spectra were performed using a SYNAPT XS High-Definition Mass Spectrometer (Waters Corporation, Manchester, UK) equipped with an electrospray ionization (ESI) source. The ions generated are transmitted through the StepWave XS ion guide to the first quadrupole (Q), then to the traveling wave ion mobility (TWIM) cell, and finally analyzed with a time-of flight (TOF) mass analyzer. UV-Visible absorption spectra were recorded on a Varian Cary 300 BIO spectrophotometer using dry and degassed dichloromethane under ambient conditions. Emission spectra were recorded on a modular Horiba FluoroLog-3 spectrofluorometer employing dry and degassed dichloromethane.

2.- Photochemically-induced synthesis of Au(III) complexes

2.1.-Synthesis of Ph₃P-Au(III) complex 8

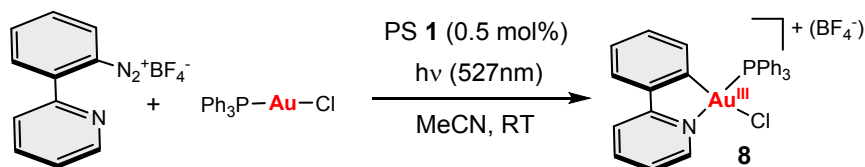


Figure S1. Synthesis of Au(III) complex 8

Complex 8 was prepared following a previous work^[3] but using complex 1 as photosensitizer: Chloro(triphenylphosphine)gold(I) (49.5 mg, 0.10 mmol, 1 equiv.), 2-(pyridin-2-yl)benzenediazonium tetrafluoroborate (27.5 mg, 0.10 mmol, 1 equiv.) and complex 1 (0.34 mg, 0.0005 mmol, 0.5%) were introduced in a high pressure Schlenk flask containing a stirring bar. The tube was evacuated and backfilled with nitrogen three times. In the absence of light, acetonitrile (1 mL) was added, and the flask was sealed. The mixture was stirred under irradiation from 5.2 mW/cm² green LEDs. After 3 h, the solvent was removed under reduced pressure and complex 8 was isolated as a pale-yellow solid in 87% yield (64.2 mg). The product was identified by comparison to previously reported data.^[3]

2.2.- Synthesis and characterization of NDI-NHC-Au(III) complex 9

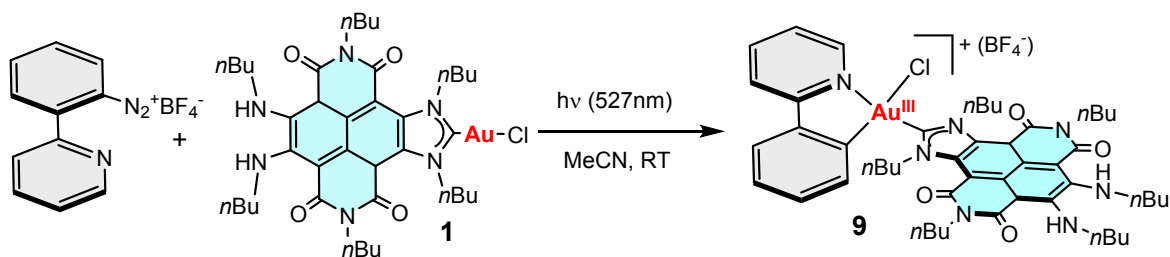


Figure S2. Synthesis of Au(III) complex 9

Complex 1 (36.2 mg, 0.04 mmol, 1 equiv.) and 2-(pyridin-2-yl)benzenediazonium tetrafluoroborate (10.8 mg, 0.04 mmol, 1 equiv.) were introduced in a high pressure Schlenk flask containing a stirring bar. The tube was evacuated and backfilled with nitrogen three times. In the absence of light, acetonitrile (4 mL) was added and the flask was sealed. The mixture was stirred under irradiation from 5.2 mW/cm² green LEDs. After 3 h, the solvent was removed under reduced pressure and complex 9 was isolated as a dark red solid almost quantitatively (40.2 mg). ¹H NMR (300 MHz, CD₃CN): δ 10.80

(t, $^3J_{\text{H-H}} = 6$ Hz, 2H, RR'NH), 9.45 (d, $^3J_{\text{H-H}} = 9$ Hz, 1H), 8.36 (t, $^3J_{\text{H-H}} = 9$ Hz, 1H), 8.24 (d, $^3J_{\text{H-H}} = 9$ Hz, 1H), 7.96 (d, $^3J_{\text{H-H}} = 9$ Hz, 1H), 7.77 (t, $^3J_{\text{H-H}} = 6$ Hz, 1H), 7.50 (t, $^3J_{\text{H-H}} = 9$ Hz, 1H), 7.18 (t, $^3J_{\text{H-H}} = 9$ Hz, 1H), 6.72 (d, $^3J_{\text{H-H}} = 9$ Hz, 1H), 5.41-5.27 (m, 2H, CH₂ imid. butyl), 5.19-5.04 (m, 2H, CH₂ imid. butyl), 4.24-4.13 (m, 4H, CH₂ naphth. butyl), 3.81-3.70 (m, 4H, CH₂ amine butyl), 1.83-1.65 (m, 6H, CH₂), 1.62-1.52 (m, 6H, CH₂), 1.50-1.40 (m, 6H, CH₂), 1.36-1.23 (m, 6H, CH₂), 1.02-0.94 (t, $^3J_{\text{H-H}} = 12$ Hz, 6H, CH₃), 0.94-0.91 (t, $^3J_{\text{H-H}} = 12$ Hz, 6H, CH₃), 0.83-0.75 (t, $^3J_{\text{H-H}} = 12$ Hz, 6H, CH₃).

¹³C{¹H} NMR (75 MHz, CDCl₃): δ 166.1 (C=O, C₉), 164.2 (C-Au^{III}, C₁), 161.7 (C=O, C₉), 160.7 (C_{carbene}-Au^{III}, C₂), 152.5 (C_q), 148.3 (CH), 144.6 (CH), 144.0 (C_q), 133.5 (CH), 133.0 (CH), 131.6 (C_q), 130.3 (CH), 127.5 (CH), 125.9 (CH), 124.9 (C_q), 122.3 (CH), 110.8 (CH), 102.4 (CH), 54.9 (CH₂, C₃), 47.4 (CH₂, C₅), 40.9 (CH₂, C₄), 34.0 (CH₂, C₆), 31.5 (CH₂, C₆), 30.4 (CH₂, C₆), 20.6 (CH₂, C₇), 20.2 (CH₂, C₇), 19.8 (CH₂, C₇), 13.7 (CH₃, C₈), 13.5 (CH₃, C₈), 13.4 (CH₃, C₈).

Electrospray MS (20 V, *m/z*): 1058.51 [M – (BF₄)]⁺ (Calcd. for [M – (BF₄)]⁺: 1058.44).

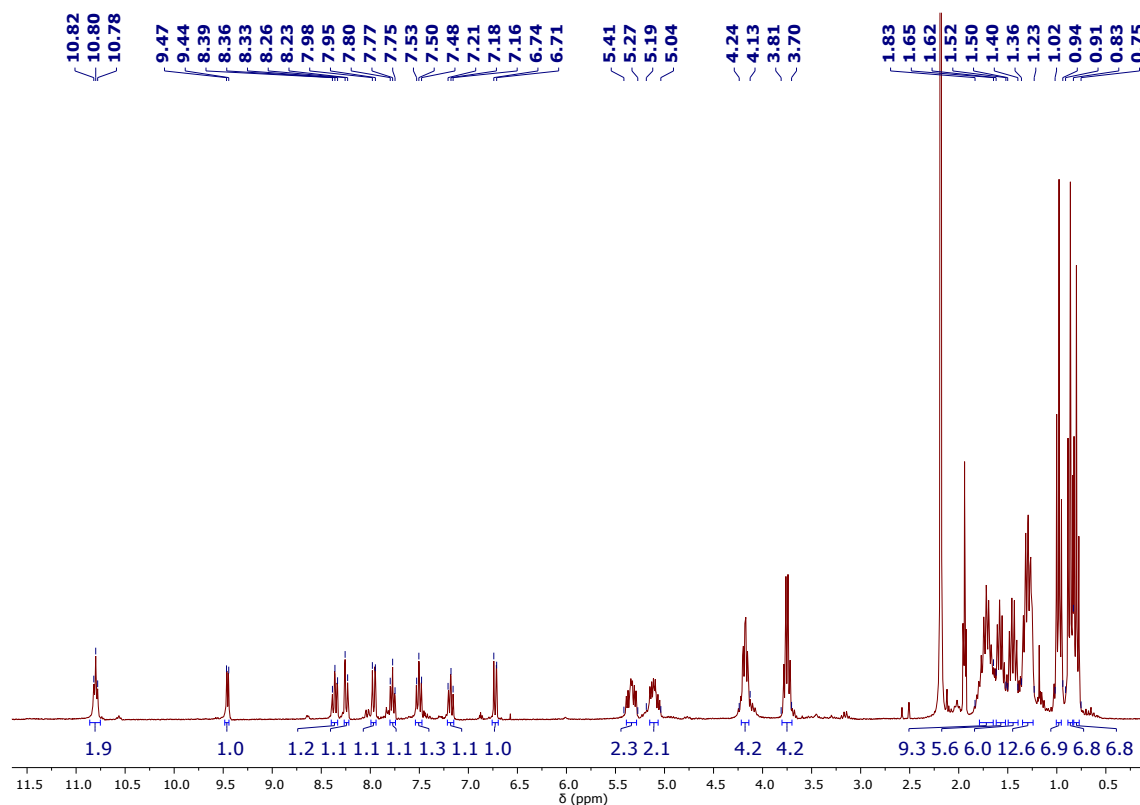


Figure S3. ¹H NMR spectrum (300 MHz, CD₃CN) of **9**

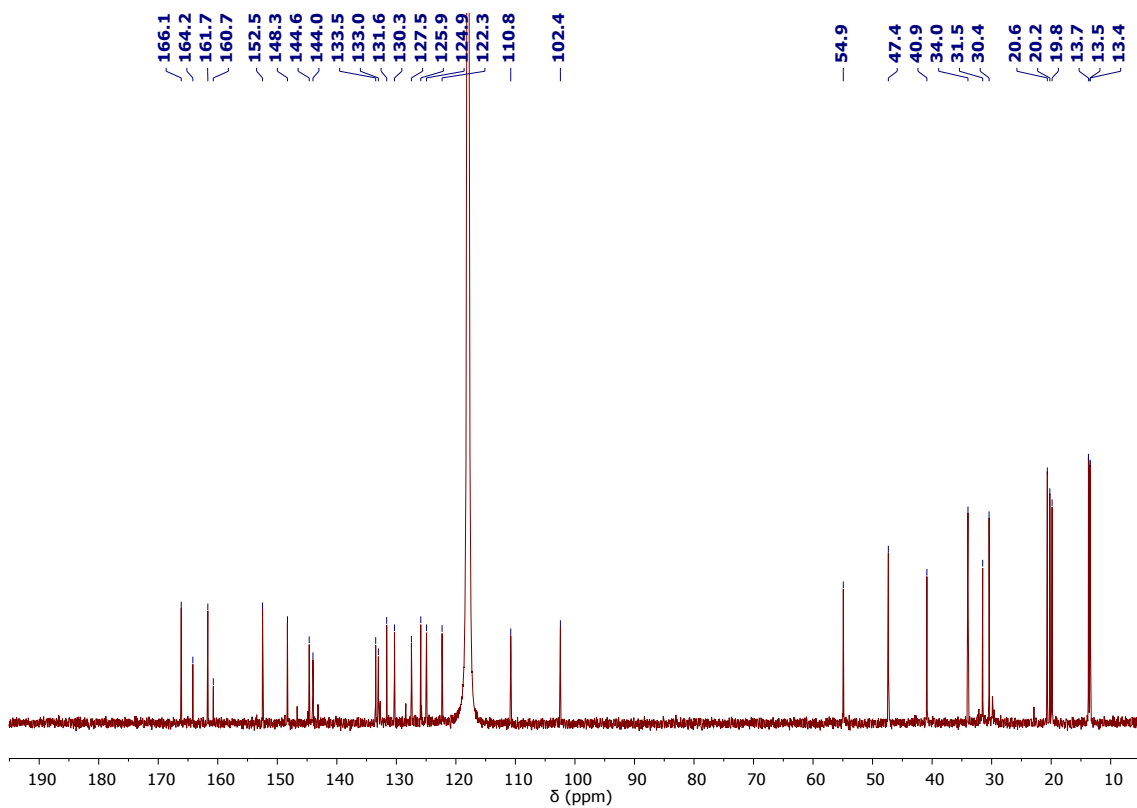


Figure S4. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum (75 MHz, CD_3CN) of **9**

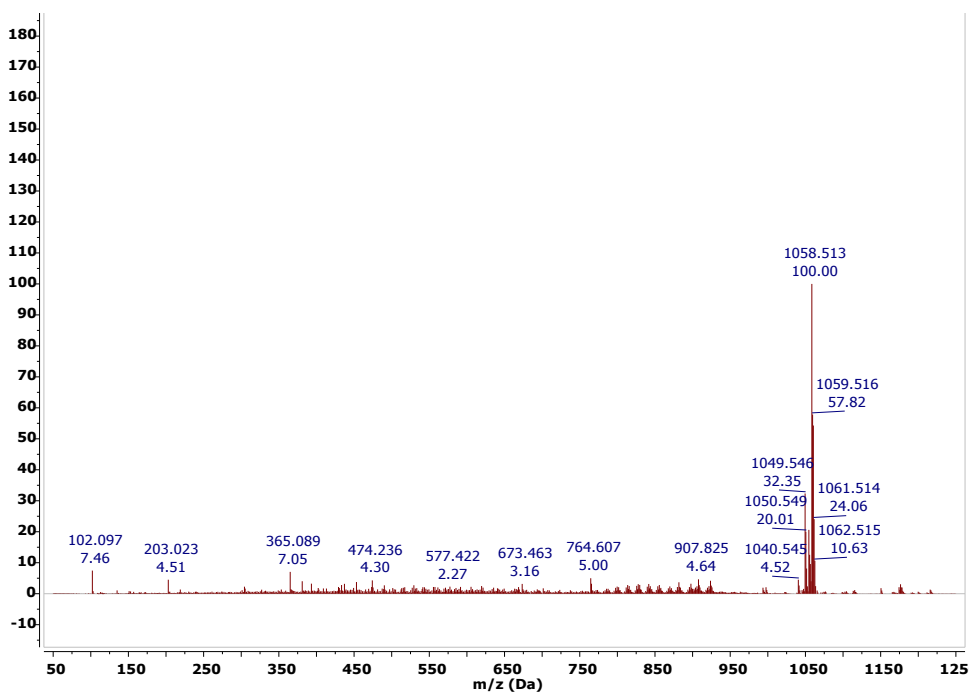


Figure S5. Mass spectrum of **9**

3. Catalytic studies

3.1.- Singlet oxygen quantum yield and endoperoxidation/hydroperoxidation

Samples were irradiated with green light ($\lambda = 527$ nm), with a 20W household RGB-LED light (5.2 mW/cm^2 incident power). The experiments were carried out placing the NMR tube or Schlenk at 15 cm of the light beam, under the exclusion from other light sources by a black box. NMR samples were protected from ambient light between the measurements. The emission spectrum of the household LED light used as light source in the photocatalyzed reaction is displayed in Figure S6.

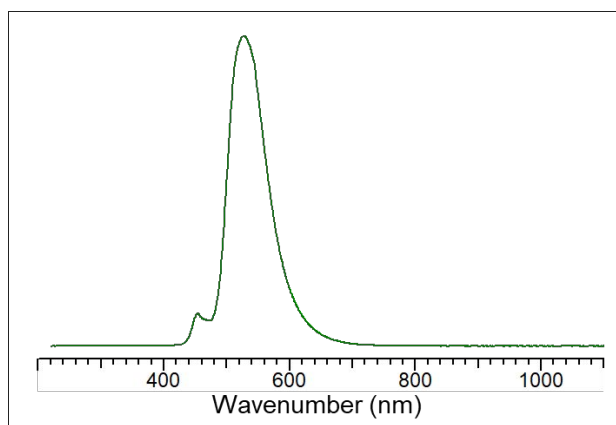


Figure S6. Emission spectrum of the green light ($\lambda_{\text{max}} = 527$ nm) used in the photocatalysis experiments.

3.1.1 Determination of the $^1\text{O}_2$ quantum yield

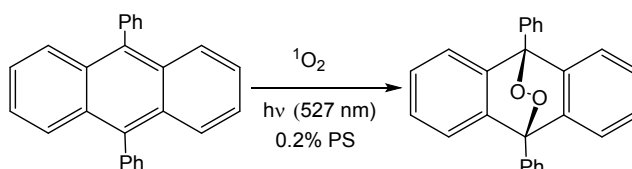


Figure S7. Photocatalytic endoperoxidation of 9,10-diphenylanthracene with $^1\text{O}_2$ using complex **1** or RB as photosensitizers.

Singlet oxygen quantum yield (Φ_{Δ}) of photosensitizer **1** was determined employing Equation S1 by comparing the reaction rates (Figure S8) of the endoperoxidation of 9,10-diphenylanthracene for complex **1** and RB used as a standard ($\Phi_{\Delta}(\text{RB}) = 0.53$), used at concentrations for which the same absorbance was observed at the irradiating wavelength. For this, the corresponding molar extinction coefficients were determined at the irradiation wavelength. The molar extinction coefficients of complex **1** and RB was calculated yielding $20.1 \text{ L mmol}^{-1}\text{cm}^{-1}$ and $42.7 \text{ L mmol}^{-1}\text{cm}^{-1}$, respectively. The reactions

were carried out using an NMR tube containing a 2 mM solution of 9,10-diphenylanthracene and 0.004 mM of RB (0.2%) or 0.0084 mM of **1** (0.42%) in oxygenated CD₃CN (0.6 mL). The samples were irradiated under O₂ atmosphere (1 atm) with a LED lamp (5.2 mW/cm²). The evolution of the reactions was determined by ¹H-NMR spectroscopy, using anisole (2 mM) as internal integration standard. The products were identified by comparison with previously reported data.^[4]

$$\Phi_{\Delta} = \Phi_{\Delta}(\text{RB}) \frac{W}{W_{\text{RB}}}$$

Equation S1. Determination of singlet oxygen quantum yield (Φ_{Δ}), where $\Phi_{\Delta}(\text{RB})$ is the singlet oxygen quantum yield for Rose Bengal (0.53), and W and W_{RB} are the reaction rates for the endoperoxidation of 9,10-diphenylanthracene in the presence of **1** and Rose Bengal, respectively.

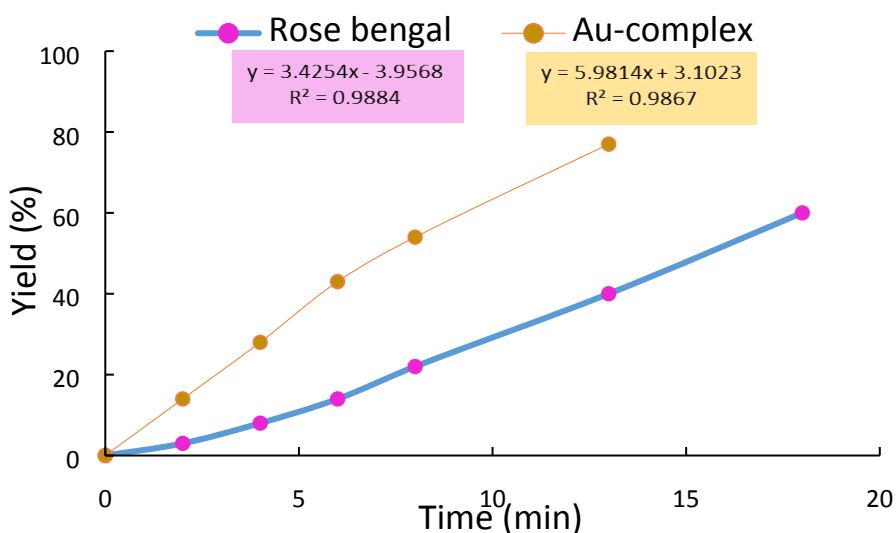
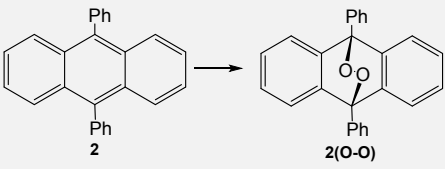
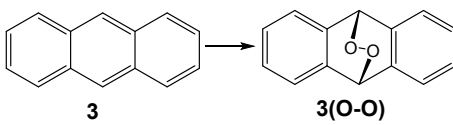
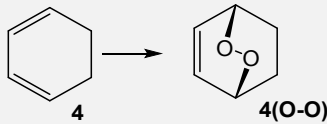
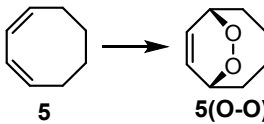
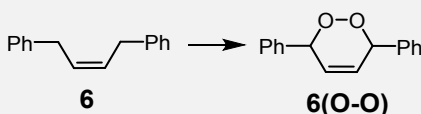
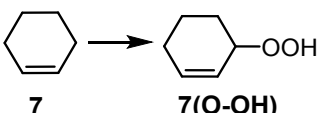


Figure S8. Time-dependent reaction profile of the endoperoxidation of 9,10-diphenylanthracene. Reactions performed under O₂ in CD₃CN under irradiation at 527 nm. The reactions were monitored by ¹H-NMR. The reactions were performed using 0.004 mM for RB (0.2%) and 0.0084 mM of complex **1**, for which the same absorbance was observed at the irradiation wavelength used in the experiment.

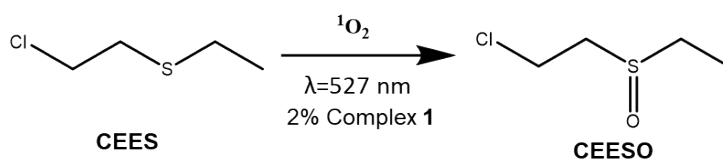
3.1.2- Peroxidation of Organic Substrates by Light-Induced Cycloaddition of ¹O₂

General procedure: All experiments were carried out using an NMR tube containing a 2 mM solution of the organic compound and 0.004-0.02 mM solution of the corresponding photosensitizer (complex **1** or RB, according to Table S1) in oxygenated CD₃CN (0.6 mL). The samples were irradiated under O₂ atmosphere (1 atm) with a LED lamp (5.2 mW/cm²). The evolution of the reactions, yields and conversions were determined by ¹H NMR spectroscopy, using anisole (2 mM) as internal standard. The products were identified by comparison to previously reported data.^[4]

Table S1. Peroxidation of organic substrates by light-induced cycloaddition of $^1\text{O}_2$.

Entry	Substrate/Product	Cat. Load.	[Substrate] (mM)	Time	Yield (%)
1		1%	0.02	10 min	>99
2		0.2%	0.004	30 min	>99
3		1%	0.02	1 h	>99
4		1%	0.02	12 h	70
5		1%	0.02	4 h	88
6		1%	0.02	24 h	66

3.1.3.- Detoxification of 2-chloroethyl ethyl sulfide (CEES)



General procedure: Complex **1** (2 mol %, 0.004 mmol), 2-chloroethyl ethyl sulfide (CEES, 23 μL , 0.2 mmol) and anisole (21 μL , 0.2 mmol) were placed together in a thick-walled Schlenk tube fitted with a Teflon cap. The tube was evacuated and filled with oxygen three times. Afterward, 1 mL of CD_3CN was added and the sample was irradiated under O_2 atmosphere (1 atm) with a LED lamp (5.2 mW/cm^2) for 20 min. The evolution of the reaction was determined by ^1H -NMR. The product (CEESO) was identified by comparison to previously reported data.^[5] Figure S9 depicts the ^1H NMR spectra of the reaction mixture at different reaction times, showing the disappearance of CEES and the formation of CEESO. The time-dependent reaction profile is plotted in Figure S10.

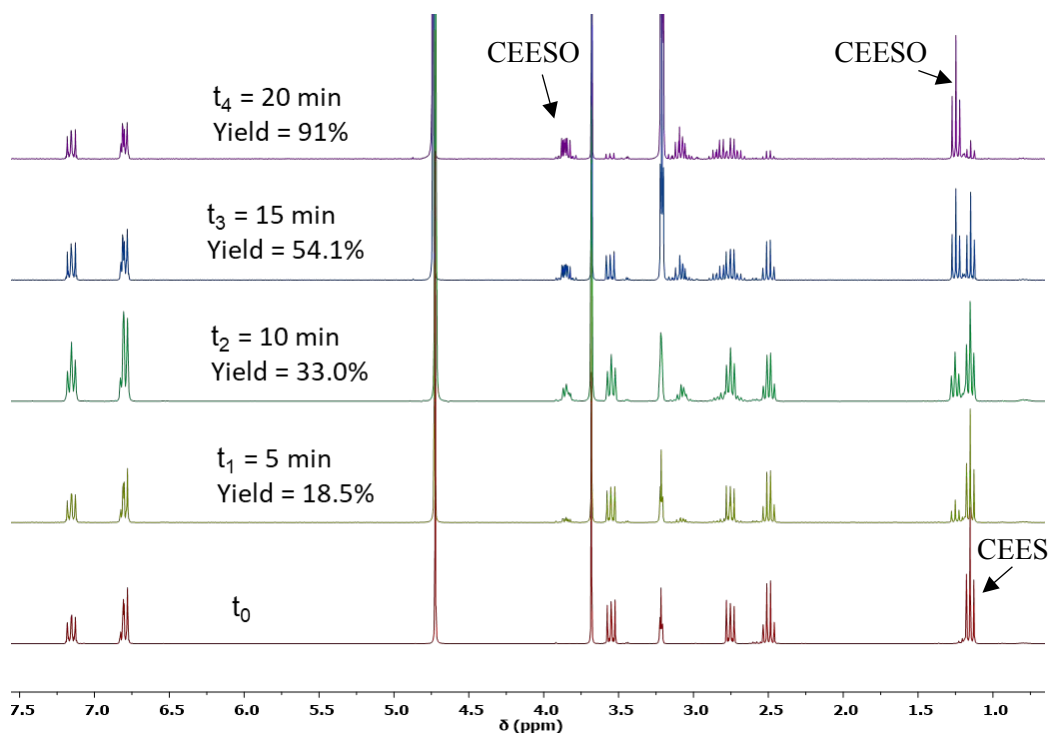


Figure S9. ^1H NMR spectra of the reaction evolution of the oxidation of CEES in CD_3CN using complex **1** as photosensitizer.

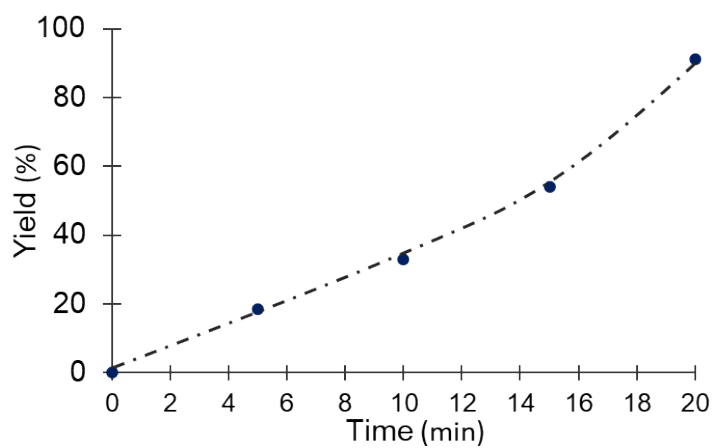
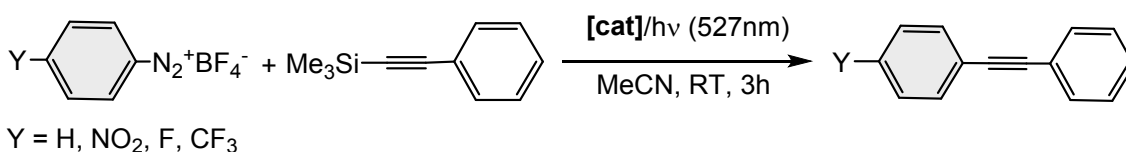


Figure S10. Time-dependent reaction profile of the oxidation of CEES using complex **1** as photosensitizer. Reactions performed under O_2 in CD_3CN under irradiation at 527 nm. The reactions were monitored by 1H -NMR.

3.2.- Metallophotoredox catalysis using complex 1

3.2.1.- Cross-coupling reactions of alkynyltrimethylsilane and aryldiazonium tetrafluoroborate salts

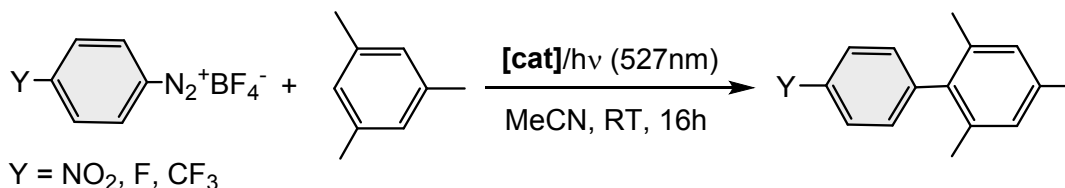


General procedure: The corresponding benzenediazonium tetrafluoroborate (0.24 mmol, 1.2 equiv.) and complex **1** (18 mg, 0.02 mmol, 10 mol %) were introduced in a high pressure Schlenk flask containing a stirring bar. At -78°C , acetonitrile (4 mL) and alkynyltrimethylsilane (39 μl , 0.2 mmol, 1 equiv.) were added to the reaction vessel. The vial was evacuated and refilled with nitrogen three times. The mixture was slowly warmed up to 23°C and stirred under irradiation from $5.2\text{ mW}/\text{cm}^2$ green LEDs. After 3 hours of reaction, the mixture was dried under vacuum and purified by flash column chromatography using a 7:3 hexane/diethyl ether mixture as eluent to give the desired products in isolated yields ranging from 60 to 74% (Table S2). All products were identified by comparison to previously reported data.^[2] For comparative purposes, the same reactions and under the same reaction conditions were simultaneously carried out in the presence of $[\text{AuCl}(\text{PPh}_3)]$ (9.9 mg, 0.02 mmol, 10 mol%) and $[\text{Ru}(\text{bpy})_3](\text{PF}_6)_2$ (3.2 mg, 0.005 mmol, 0.5 mol%) (entries 2, 4, 6 and 8).

Table S2. Dual gold photoredox cross-coupling reactions of aryldiazonium tetrafluoroborates and trimethylsilyl ethynylbenzene.

Entry	Y	Catalyst	Yield (%)
1	H	1	60
2	H	$[\text{AuCl}(\text{PPh}_3)] + [\text{Ru}(\text{bpy})_3]^{2+}$	58
3	NO ₂	1	74
4	NO ₂	$[\text{AuCl}(\text{PPh}_3)] + [\text{Ru}(\text{bpy})_3]^{2+}$	59
5	F	1	66
6	F	$[\text{AuCl}(\text{PPh}_3)] + [\text{Ru}(\text{bpy})_3]^{2+}$	60
7	CF ₃	1	60
8	CF ₃	$[\text{AuCl}(\text{PPh}_3)] + [\text{Ru}(\text{bpy})_3]^{2+}$	52

3.2.2.-Cross-coupling reactions of mesitylene and aryldiazonium tetrafluoroborate salts



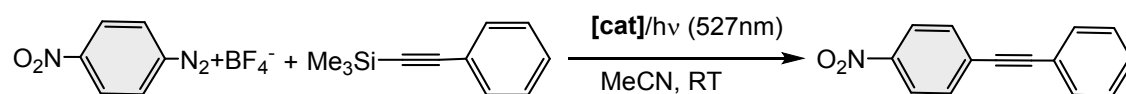
General procedure: The corresponding benzenediazonium tetrafluoroborate (0.1 mmol, 1 equiv.) and complex **1** (9 mg, 0.01 mmol, 10 mol %) were introduced in a high pressure Schlenk flask containing a stirring bar. At -78°C , acetonitrile (4 mL) and mesitylene (83 μL , 0.6 mmol, 6 equiv.) were added. The mixture was degassed using 3 freeze-pump-thaw cycles. The mixture was slowly warmed up to 23°C and stirred under irradiation from 5.2 mW/cm^2 green LEDs. After 16 hours of reaction, the mixture was dried under vacuum. All yields were calculated by $^1\text{H NMR}$ by using 1,3,5-trimethoxybenzene as integration standard (Table S3). All products were identified by comparison to previously reported data.^[6] For comparative purposes, the same reactions and under the same reaction conditions were simultaneously carried out in the presence of $[\text{AuCl}(\text{PPh}_3)]$ (4.5 mg, 0.01 mmol, 10 mol%) and $[\text{Ru}(\text{bpy})_3](\text{PF}_6)_2$ (1.6 mg, 0.0005 mmol, 0.5 mol%) (entries 2, 4 and 6).

Table S3. Dual gold photoredox coupling of aryldiazonium tetrafluoroborates and mesitylene.

Entry	Y	catalyst	Yield (%)
1	NO ₂	1	67
2	NO ₂	$[\text{AuCl}(\text{PPh}_3)] + [\text{Ru}(\text{bpy})_3]^{2+}$	57
3	F	1	90
4	F	$[\text{AuCl}(\text{PPh}_3)] + [\text{Ru}(\text{bpy})_3]^{2+}$	79
5	CF ₃	1	34
6	CF ₃	$[\text{AuCl}(\text{PPh}_3)] + [\text{Ru}(\text{bpy})_3]^{2+}$	21

3.2.3.- Time-dependent reaction profiles of cross-coupling reactions

a) coupling of aryldiazonium tetrafluoroborates with trimethylsilylethynylbenzene



General procedure: 4-Nitrobenzenediazonium tetrafluoroborate (0.03 mmol, 1 equiv.) and complex **1** (2.25 μ mol, 7.5 mol %) were introduced in an NMR tube. CD_3CN (0.5 mL) and alkynyltrimethylsilane (5.9 μ l, 0.03 mmol, 1 equiv.) previously deoxygenated, were added to the reaction. The NMR tube contained a coaxial tube with 1,3,5-trimethoxybenzene, which was used as internal integration standard. The mixture was stirred under irradiation with a 20 W white LEDs for the appropriate time. The evolution of the reactions was determined by NMR (Figure S12). For comparative purposes, the same reactions and under the same reaction conditions were simultaneously carried out in the presence of $[\text{AuCl}(\text{PPh}_3)]$ (2.25 μ mol, 7.5 mol %) and $[\text{Ru}(\text{bpy})_3](\text{PF}_6)_2$ (0.1125 μ mol, 0.375 mol%) (Figure S13). The time-dependent reaction profiles of the coupling of p-nitrophenyldiazonium tetrafluoroborate with trimethylsilylethynylbenzene is displayed in Figure S11.

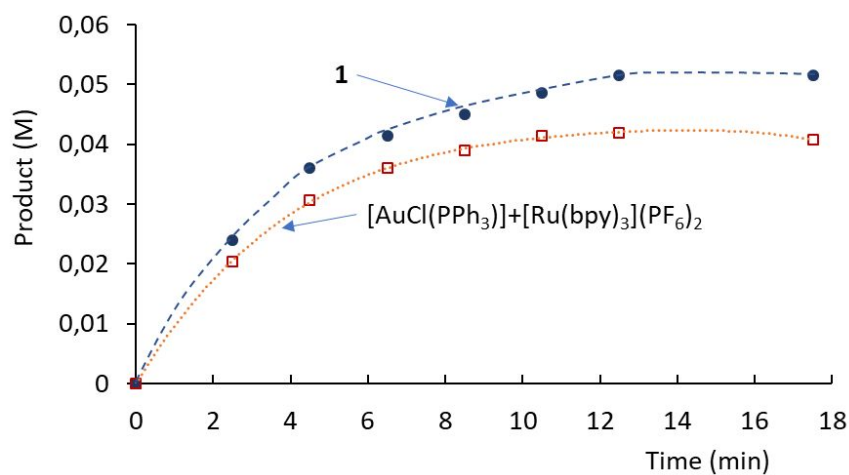


Figure S11. Time-dependent reaction profiles of the coupling of p-nitrophenyldiazonium tetrafluoroborate with trimethylsilylethynylbenzene with complex **1** (blue dots) and $[\text{AuCl}(\text{PPh}_3)] + [\text{Ru}(\text{bpy})_3](\text{PF}_6)_2$ (orange squares). The reactions were carried out in CD_3CN at room temperature using a 20W LED white lamp. The initial concentration of both substrates was 0.06M.

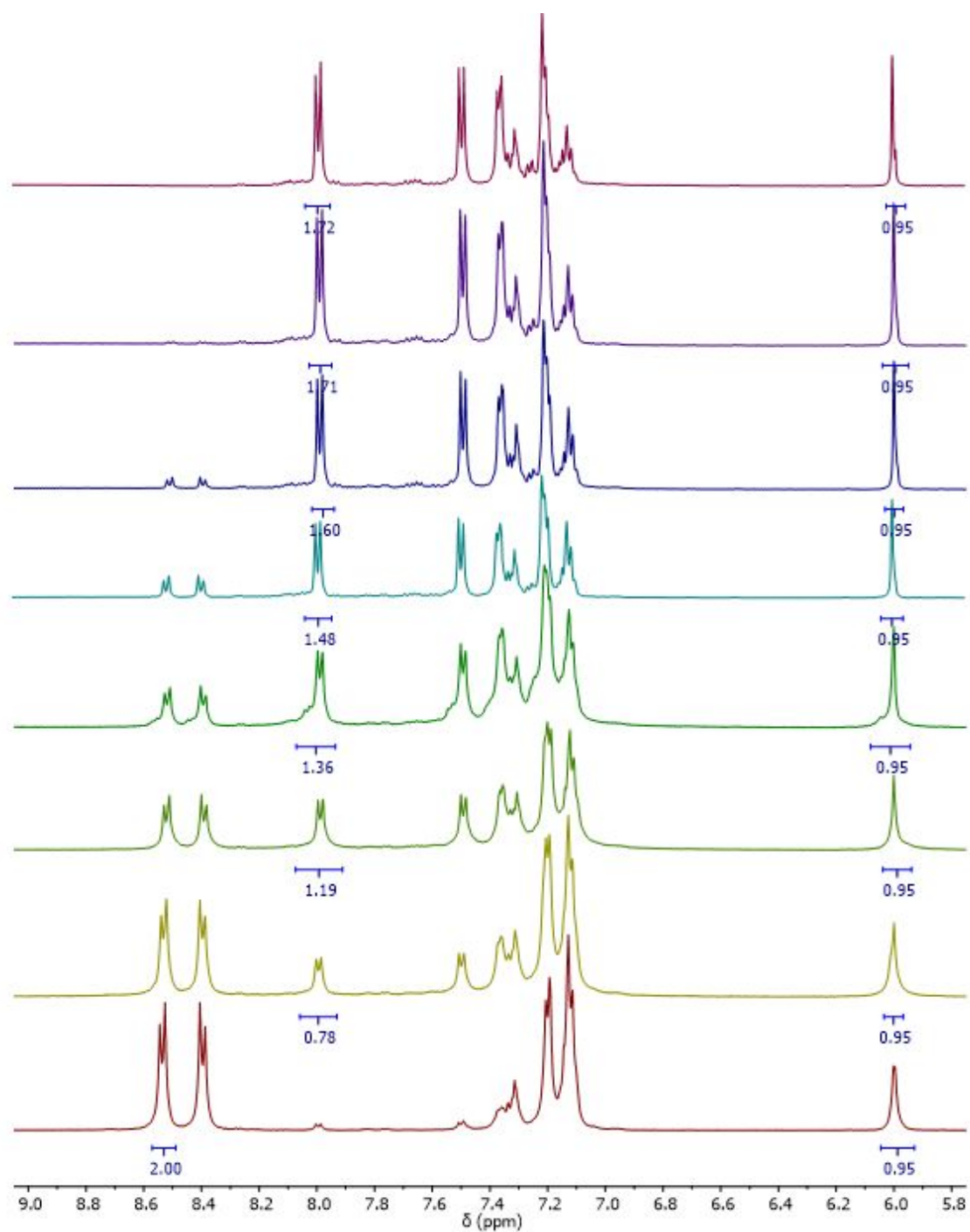


Figure S12. ¹H NMR spectra of the reaction evolution of the coupling of aryldiazonium tetrafluoroborates and trimethylsilylethynylbenzene in CD₃CN using complex **1** (7.5 mol%) as photosensitizer. The reactions were carried out in CD₃CN at room temperature using a 20W LED white lamp. The initial concentration of both substrates was 0.06M. The signal displayed at 6.0 ppm is due to 1,3,5-trimethoxybenzene, which was used as integration standard.

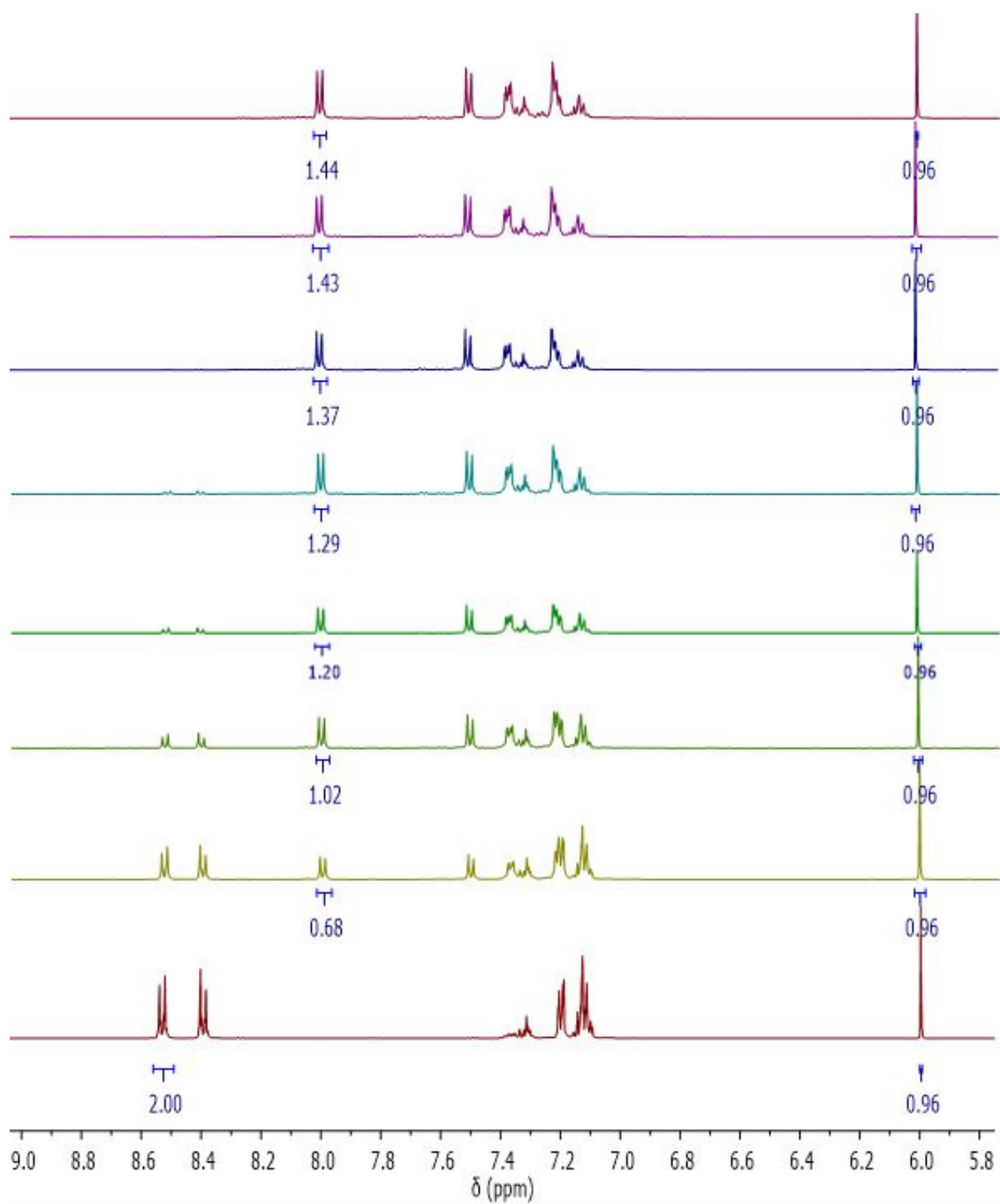
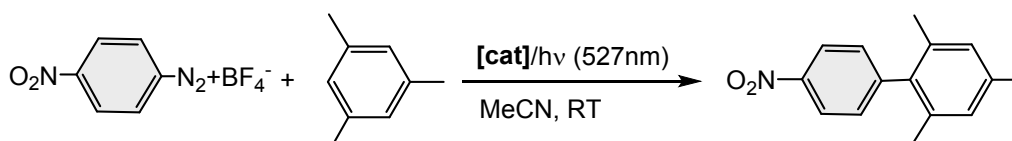


Figure S13. ^1H NMR spectra of the reaction evolution of the coupling of aryldiazonium tetrafluoroborates with trimethylsilylethynylbenzene in CD_3CN using as $[\text{AuCl}(\text{PPh}_3)]$ (7.5 mol%) and $[\text{Ru}(\text{bpy})_3](\text{PF}_6)_2$ (0.0375 mol%). The reactions were carried out in CD_3CN at room temperature using a 20W LED white lamp. The initial concentration of both substrates was 0.06M. The signal displayed at 6.0 ppm is due to 1,3,5-trimethoxybenzene, which was used as integration standard.

b) Coupling of *p*-nitrophenyldiazonium tetrafluoroborate with mesitylene



General procedure: 4-Nitrobenzenediazonium tetrafluoroborate (0.03 mmol, 1 equiv.) and complex **1** (2.25 μmol , 7.5 mol %) were introduced in an NMR tube. CD_3CN (0.5 mL) and mesitylene (24.2 μl , 0.18 mmol, 6 equiv.) previously deoxygenated, were added to the reaction. The NMR tube contained a coaxial tube with 1,3,5-trimethoxybenzene, which was used as internal integration standard. The mixture was stirred under irradiation with a 20 W white LEDs for the appropriate time. The evolution of the reactions was determined by NMR spectroscopy (Figure S15). For comparative purposes, the same reactions and under the same reaction conditions were simultaneously carried out in the presence of $[\text{AuCl}(\text{PPh}_3)]$ (2.25 μmol , 7.5 mol %) and $[\text{Ru}(\text{bpy})_3](\text{PF}_6)_2$ (0.1125 μmol , 0.375 mol%) (Figure S16). The time-dependent reaction profiles of the coupling of *p*-nitrophenyldiazonium tetrafluoroborate with trimethylsilylethynylbenzene is displayed in Figure S14.

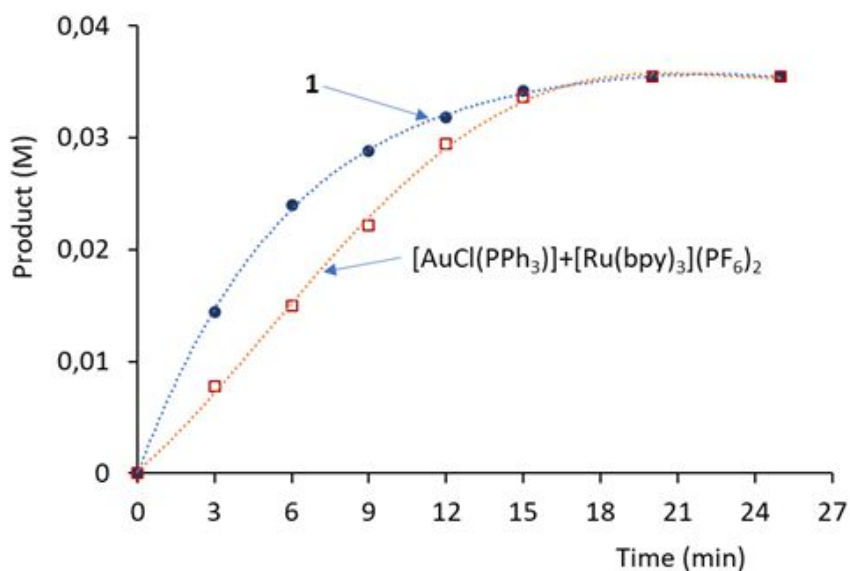


Figure S14. Time-dependent reaction profiles of the coupling of *p*-nitrophenyldiazonium tetrafluoroborate with mesitylene with complex **1** (blue dots) and $[\text{AuCl}(\text{PPh}_3)] + [\text{Ru}(\text{bpy})_3](\text{PF}_6)_2$ (orange squares). The reactions were carried out in CD_3CN at room temperature using a 20W LED white lamp.

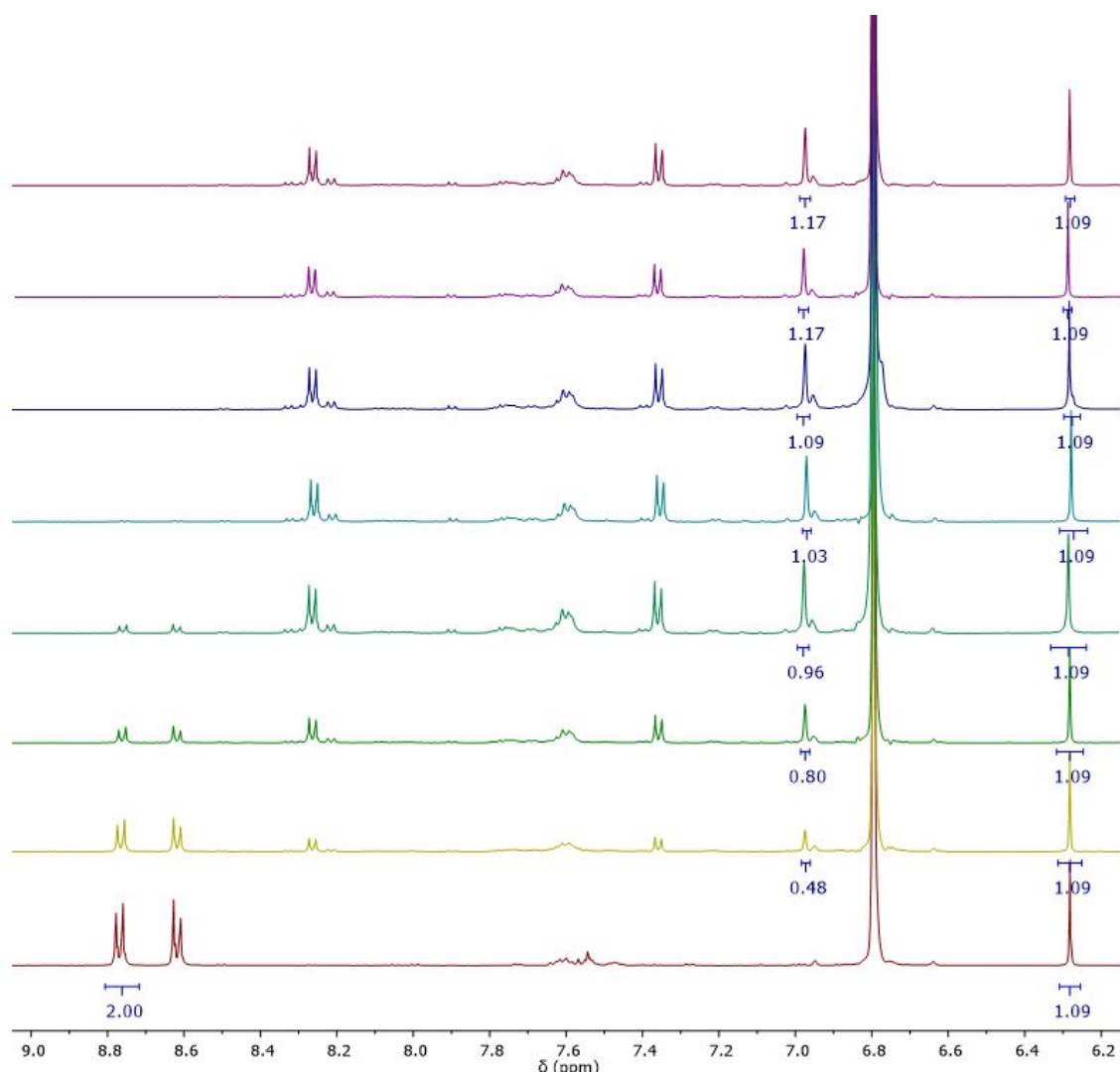


Figure S15. ¹H NMR spectra of the reaction evolution of the coupling of aryldiazonium tetrafluoroborates and mesitylene in CD₃CN using complex **1** (7.5 mol%) as photosensitizer. The reactions were carried out in CD₃CN at room temperature using a 20W LED white lamp. The signal displayed at 6.0 ppm is due to 1,3,5-trimethoxybenzene, which was used as integration standard.

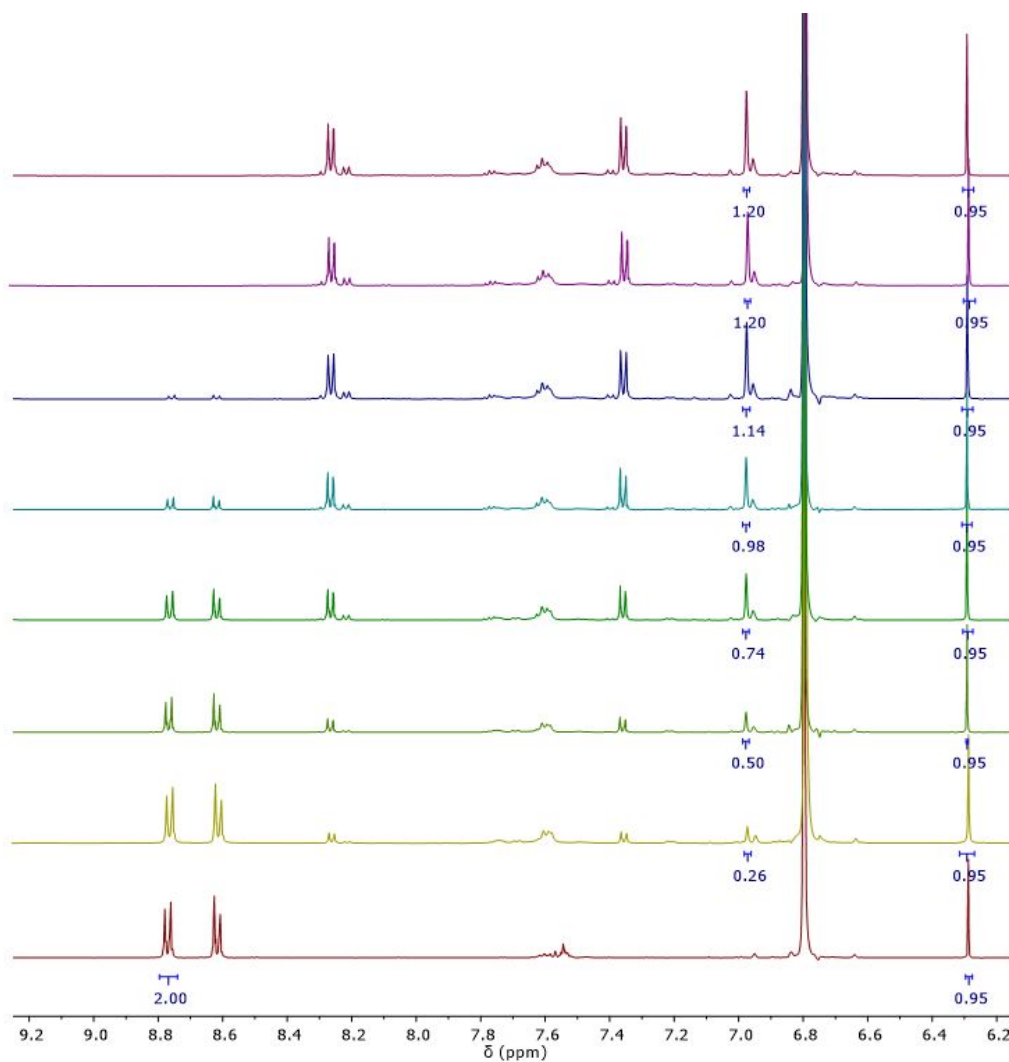


Figure S16. ^1H NMR spectra of the evolution of the coupling of aryldiazonium tetrafluoroborates and mesitylene in CD_3CN , using as $[\text{AuCl}(\text{PPh}_3)]$ (7.5 mol%) and $[\text{Ru}(\text{bpy})_3](\text{PF}_6)_2$ (0.0375 mol%). The reactions were carried out in CD_3CN at room temperature using a 20W LED white lamp. The signal displayed at 6.0 ppm is due to 1,3,5-trimethoxybenzene, which was used as integration standard.

4. Electrochemical measurements

Electrochemical studies were carried out by using an Autolab Potentiostat, Model PGSTAT101 controlled with NOVA 2.1.4 software. In all experiments, $[N(nBu)_4][PF_6]$ (0.25 M in dry and deoxygenated CH_3CN) was used as the supporting electrolyte with an analyte concentration of 1 mM. Cyclic voltammetry was performed in a cell, under N_2 atmosphere and with disk glassy carbon working electrode, platinum counter electrode, and a silver wire pseudoreference electrode. All scans were referenced to the ferrocenium/ferrocene (Fc^+/Fc) couple at 0 V. Ohmic drop was minimized by minimizing the distance between the working and reference electrodes. The residual ohmic drop was estimated by positive feedback and compensated at 95%.

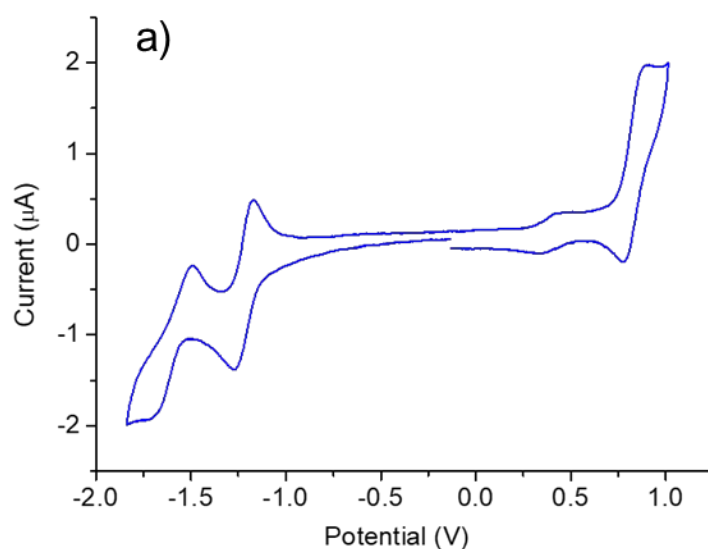


Figure S17. Cyclic voltammograms of complex **1** referenced to the ferrocenium/ferrocene (Fc^+/Fc) couple at 0V.

5. Photophysical analysis

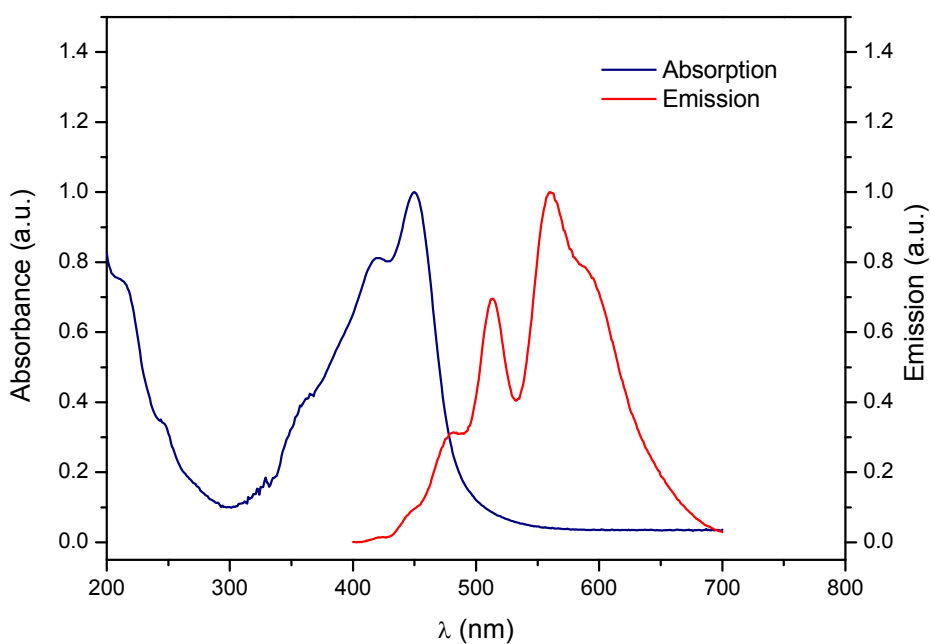


Figure S18. UV-Vis spectra (blue line) and emission spectra (red line) excited at 375 nm of salt A recorded in CH_2Cl_2 at a concentration of 50 μM .

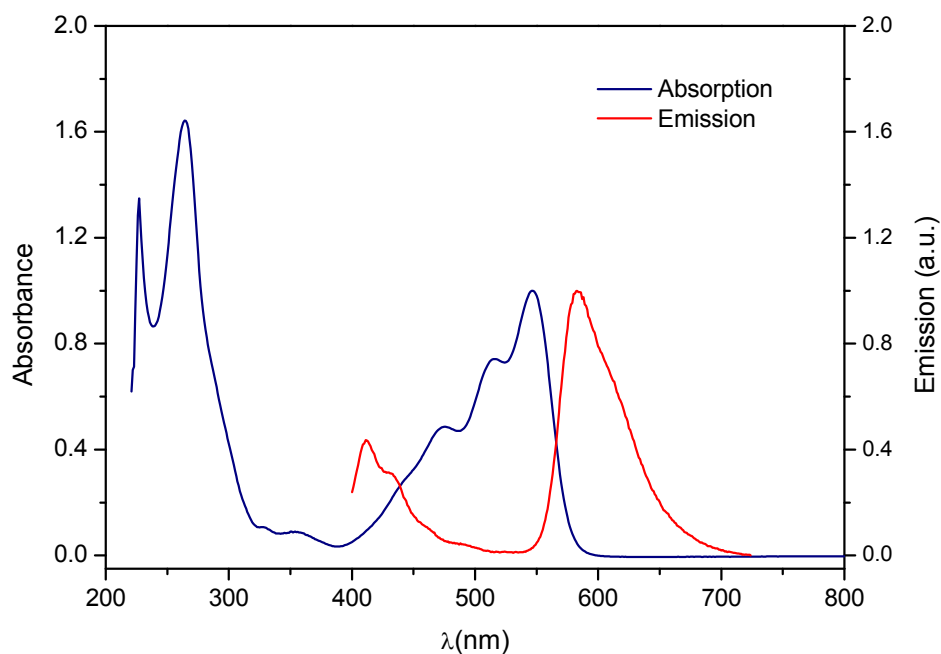


Figure S19. UV-Vis spectra (blue line) and emission spectra (red line) excited at 375 nm of complex 1 recorded in CH_2Cl_2 at a concentration of 50 μM .

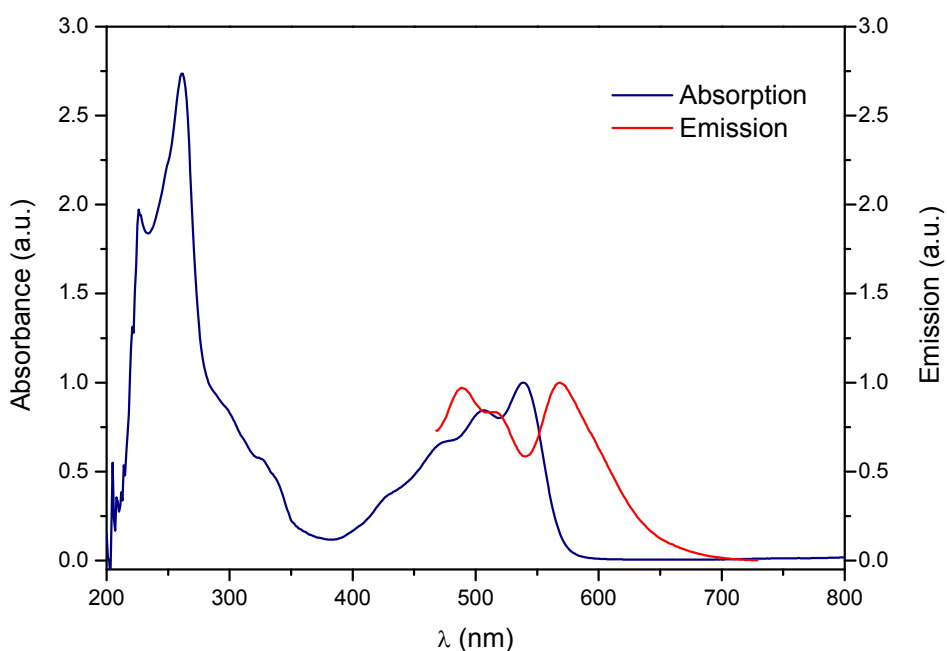


Figure S20. UV-Vis spectra (blue line) and emission spectra (red line) excited at 375 nm of complex **9** recorded in CH_2Cl_2 at a concentration of 50 μM .

6. References

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