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Supporting Information

Aqueous Platinum(II)-Cage-Based Light-Harvesting System for Photocatalytic Cross-Coupling Hydrogen Evolution Reaction

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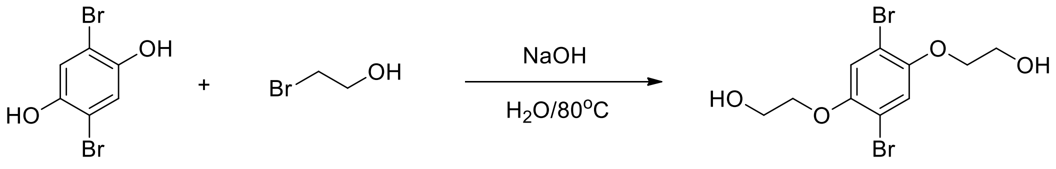
1. Materials and methods

All reagents and deuterated solvents were commercially available and used without further purification. Compounds **1**[S1] and **3**[S2] were prepared according to the published procedure. NMR spectra were recorded on a Bruker Avance 400 MHz or 600 MHz spectrometer. ^{1}H and ^{13}C NMR chemical shifts are recorded relative to residual solvent signals. ^{31}P{^{1}H} NMR chemical shifts are referenced to an external unlocked sample of 85% H_3PO_4 (δ 0.0). Mass spectra were recorded on a Micromass Quattro II triple-quadrupole mass spectrometer using electrospray ionization with a MassLynx operating system. The UV-vis experiments were conducted on a Lambd 950 absorption spectrophotometer. The fluorescent experiments were conducted on a Edinburgh FLS9 fluorescence spectrophotometer. Transmission electron microscopy (TEM) investigations were carried out on a JEOL JEM-F200(HR) instrument. Confocal laser scanning microscopy (CLSM) was performed with a Zeiss LSM 710 Confocal Microscope using a 63× objective. Dynamic light scattering experiments were performed using a Malvern Nano-ZS instrument with a He-Ne laser (633 nm) and 173° collecting optics. The 300 W xenon lamp was used for photoreaction and the optical power meter was supplied by Beijing Perfect Light Co. Ltd. The light power is 150 mW/cm^2.

2. Synthetic Procedures and Characterization Data

*2.1 Synthesis of compound **10***



8 **9** **10**, 92%

NaOH (1.20 g, 30 mmol) was added to a suspension of 2,5-dibromobenzene-1,4-diol (compound **8**, 2.67 g, 10 mmol) in water. The solution was heated at reflux for 2 h under nitrogen. Then 2-bromoethanol (compound **9**, 3.75 g, 30 mmol) was added dropwise to the system. The resulting solution was refluxed for another 3h. After cooling down to room temperature, off white precipitate was formed. The mixture was filtered and the filter cake was washed with portions of water for 4 times to give compound **10** (3.27 g, 92%) as an off-white solid. ^{1}H NMR (600 MHz, DMSO-d_6): 7.38 (s, 2H), 4.86 (t, J = 5.2 Hz, 2H), 4.03 (t, J = 5.2 Hz, 4H), 3.70 (m, 4H). The ^{1}H NMR spectra of **10** matches well with reported results.[S3]

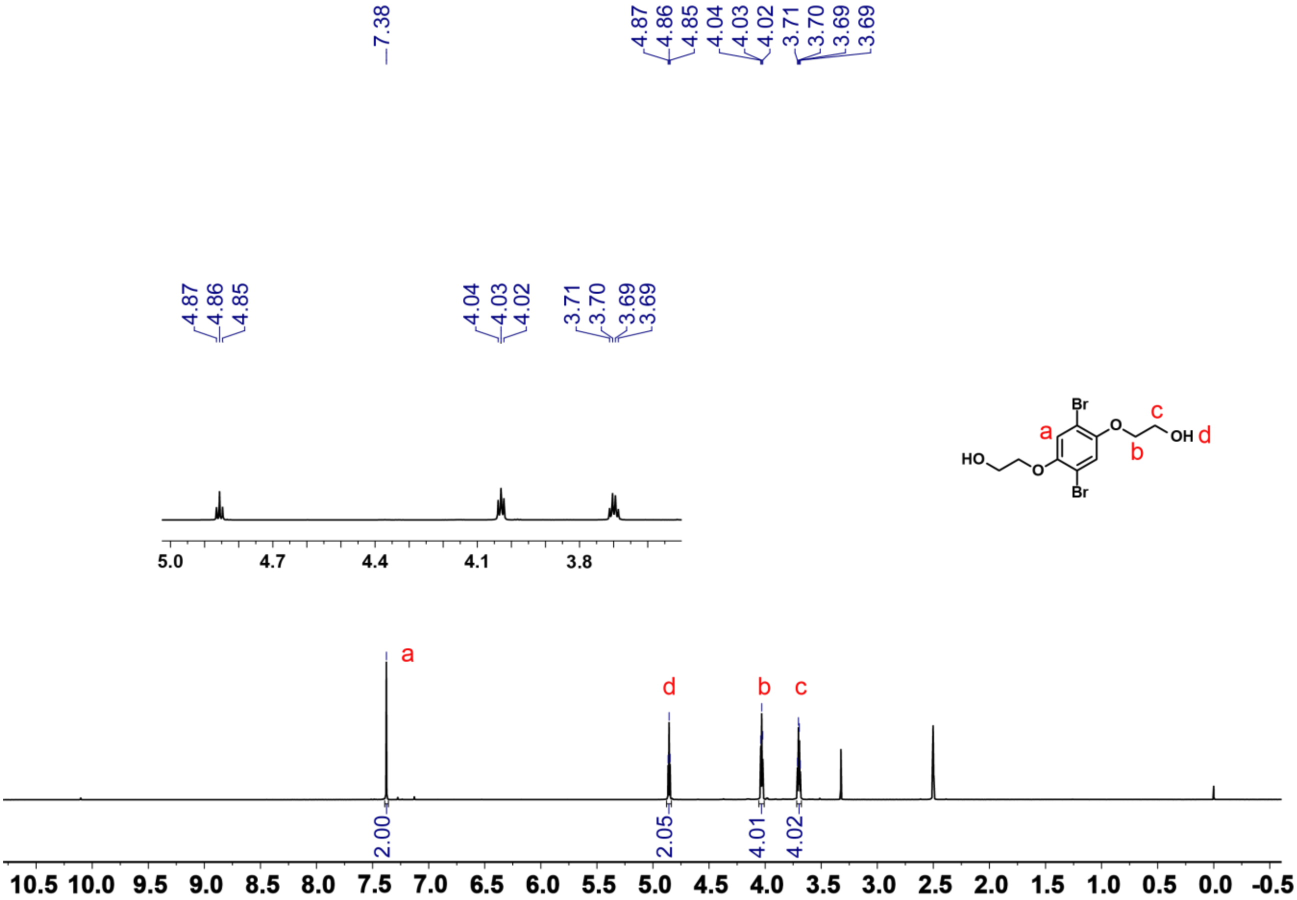
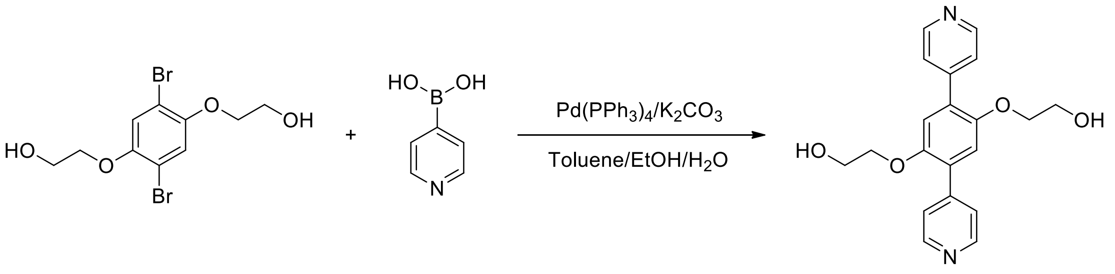


Figure S1. ^{1}H NMR spectrum (600 MHz, DMSO-d_6, 295 K) recorded for **10**.

*2.2 Synthesis of compound **2a***



10 **11** **2a**, 88%

Compound **10** (356 mg, 1.00 mmol), 4-pyridylboronic acid (compound **11**, 369 mg, 3.00 mmol), $Pd(PPh_3)_4$ (116 mg, 0.1 mmol) and K_2CO_3 (553 mg, 4 mmol) were dissolved in toluene/ethanol/water (*v*/*v*/*v* = 4:1:1, 40 mL) in a 100 mL Schlenk flask. Then the mixture was cooled by liquid nitrogen, degassed and purged with nitrogen for three times. The reaction mixture was stirred at 90℃ for 40 h under nitrogen. After cooling, the product was concentrated to give a crude product which was further purified by washing with ethanol (15 mL × 3) to afford compound **2a** (310 mg, 88%) as an off-white solid. ^{1}H NMR (400 MHz, DMSO-d_6): 8.63 (dd, J = 4.5, 1.6 Hz, 4H), 7.72 (dd, J = 4.5, 1.6 Hz, 4H), 7.23 (s, 2H), 4.87 (t, J = 5.3 Hz, 2H), 4.11 (t, J = 4.9 Hz, 4H), 3.69 (m, 4H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 150.48, 149.88, 145.36, 128.55, 124.58, 116.11, 71.37, 60.01. ESI-HR-MS: *m/z* 353.1492 [**2a** + H]$^+$, calcd. for $[C_{20}H_{21}N_2O_4]^+$, 353.1496.

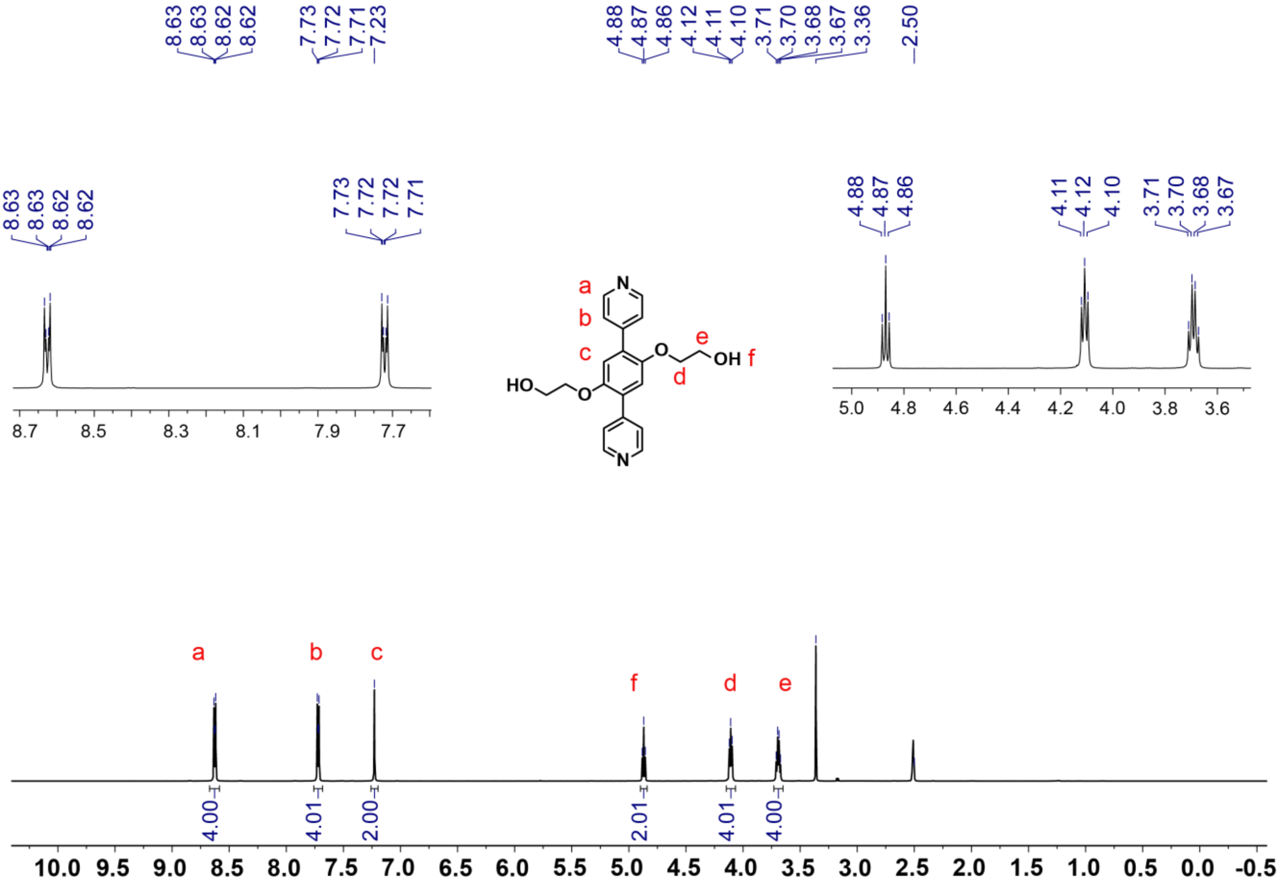


Figure S2. ^{1}H NMR spectrum (400 MHz, DMSO-d_6, 295 K) recorded for **2a**.

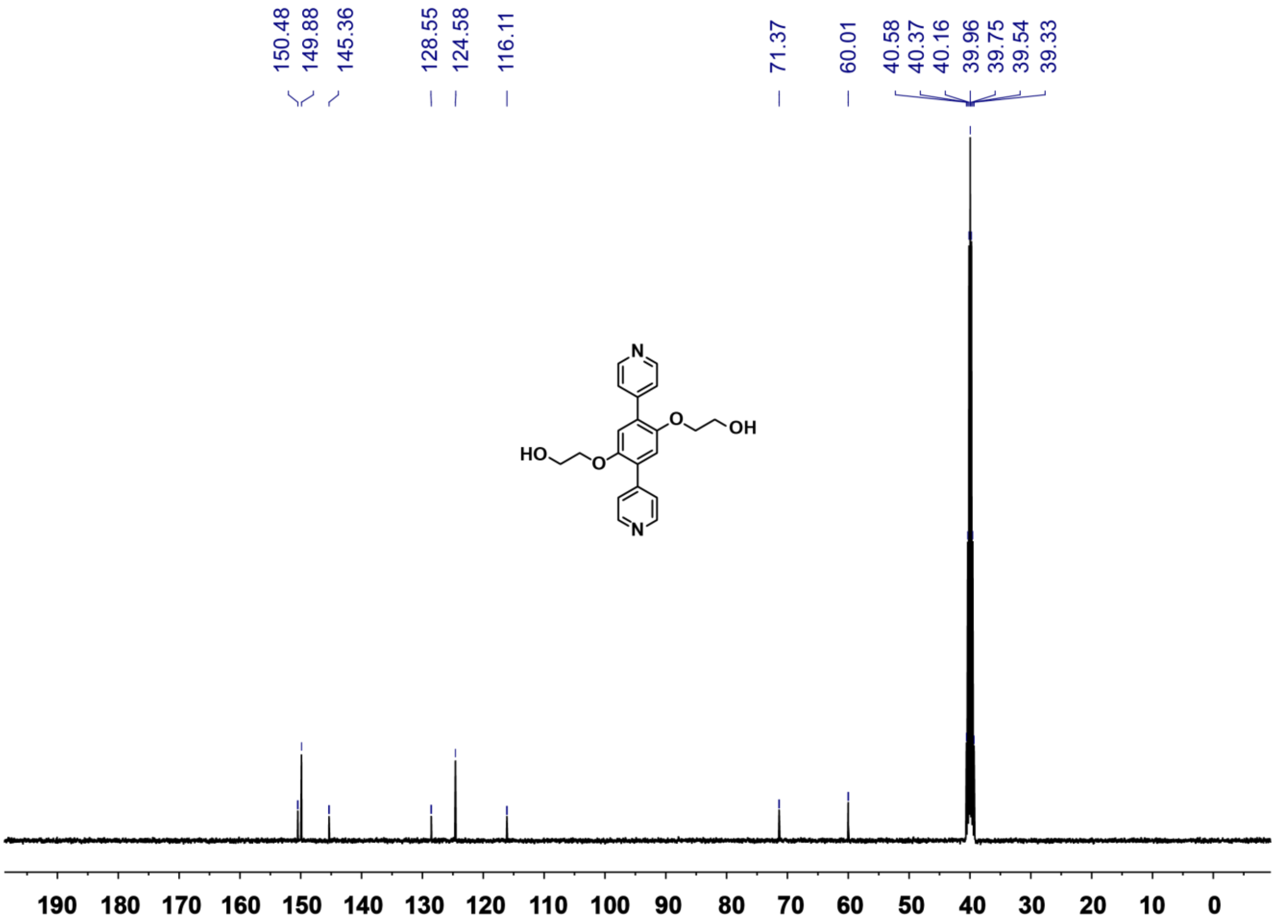


Figure S3. ^{13}C NMR spectrum (100 MHz, DMSO-d_6, 295 K) recorded for **2a**.

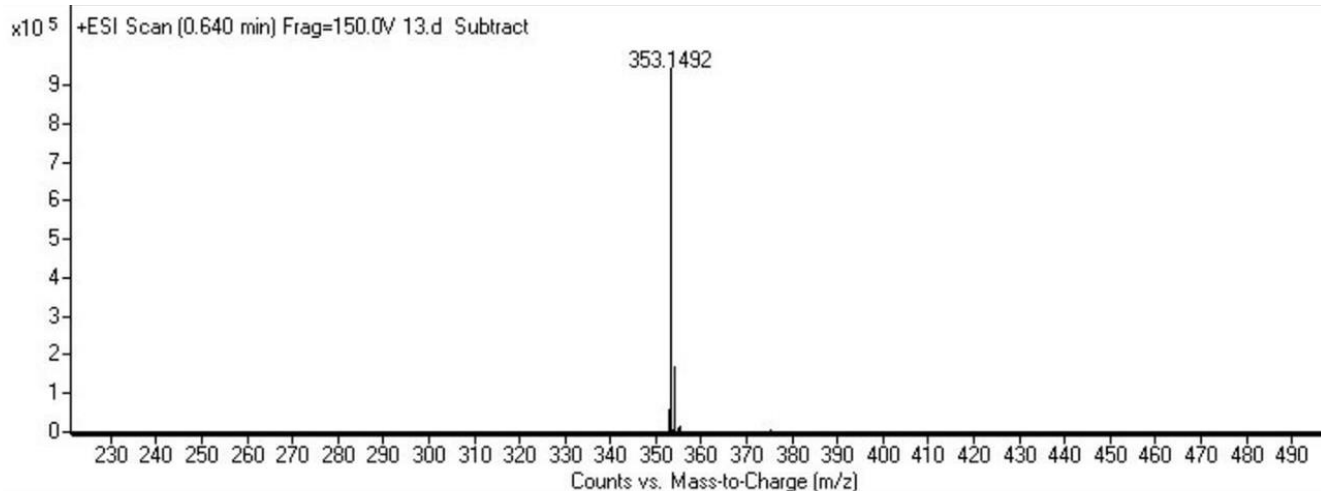
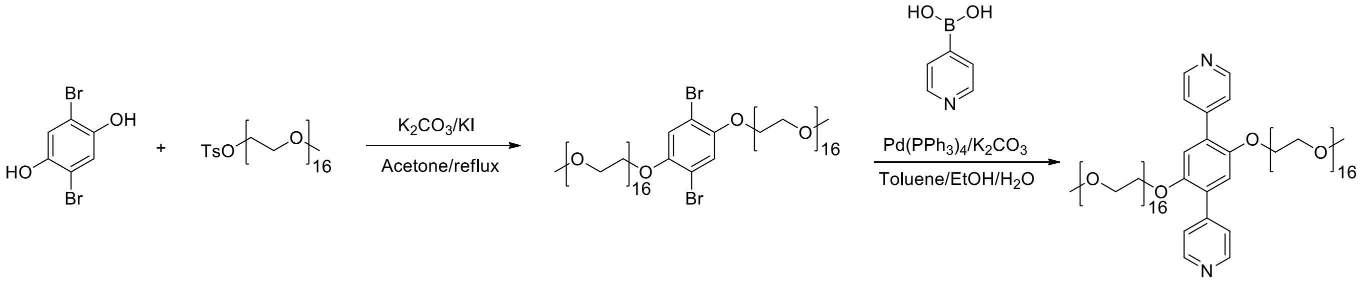


Figure S4. ESI-HR-MS spectrum of **2a**.

2.3 Synthesis of compound ***2b***



12 **13** **14**, 76% **2b**, 82%

Compound **12** (535 mg, 2.00 mmol), tosylated PEG-750 (4.073 g, 6.00 mmol), K_2CO_3 (1.10 g, 8.00 mmol) and KI (17 mg, 0.1 mmol) were dissolved in acetone (750 mL) and the reaction mixture was heated at reflux for 15 h under nitrogen. Then the system was cooled down and the solvent was removed. H_2O (100 mL) was added and the mixture was extracted by dichloromethane (50 mL × 3). The organic phase was collected, dried over anhydrous Na_2SO_4 and then concentrated. The residue (2.687 g, 76%) was directly used for next step. Compound **14** (1.764 g, 1.00 mmol), 4-pyridylboronic acid (compound **11**, 369 mg, 3.00 mmol), $Pd(PPh_3)_4$ (116 mg, 0.1 mmol) and K_2CO_3 (553 mg, 4 mmol) were dissolved in toluene/ethanol/water ($v/v/v$ = 4:1:1, 40 mL) in a 100 mL Schlenk flask. Then the mixture was cooled by liquid nitrogen, degassed and purged with nitrogen for three times. The reaction mixture was stirred at 90℃ for 40 h under nitrogen. After cooling, the solvent was removed. H_2O (100 mL) was added and the mixture was extracted by dichloromethane (50 mL × 3). The organic phase was collected, dried over anhydrous Na_2SO_4 and then concentrated to give a crude product which was purified by flash column chromatography with CH_2Cl_2:CH_3OH (15:1 to 10:1, v/v) as the eluent to afford compound **2b** (1.443 g, 82%) as a pale-yellow oil. ^{1}H NMR (400 MHz, DMSO-d_6, 295 K): 8.63 (d, J = 6.0 Hz, 4H), 7.72 (d, J = 6.0 Hz, 4H), 7.25 (s, 2H), 4.26–4.18 (m, 4H), 3.74–3.69 (m, 4H), 3.58–3.40 (m, 122H), 3.24 (s, 6H). ^{13}C NMR (100 MHz, DMSO-d_6, 295 K) δ 150.38, 149.82, 145.24, 128.54, 124.57, 116.01, 71.75, 70.56–69.92 (m), 69.38, 68.98, 58.50, 40.58, 40.37, 40.16, 39.95, 39.75, 39.54, 39.33.

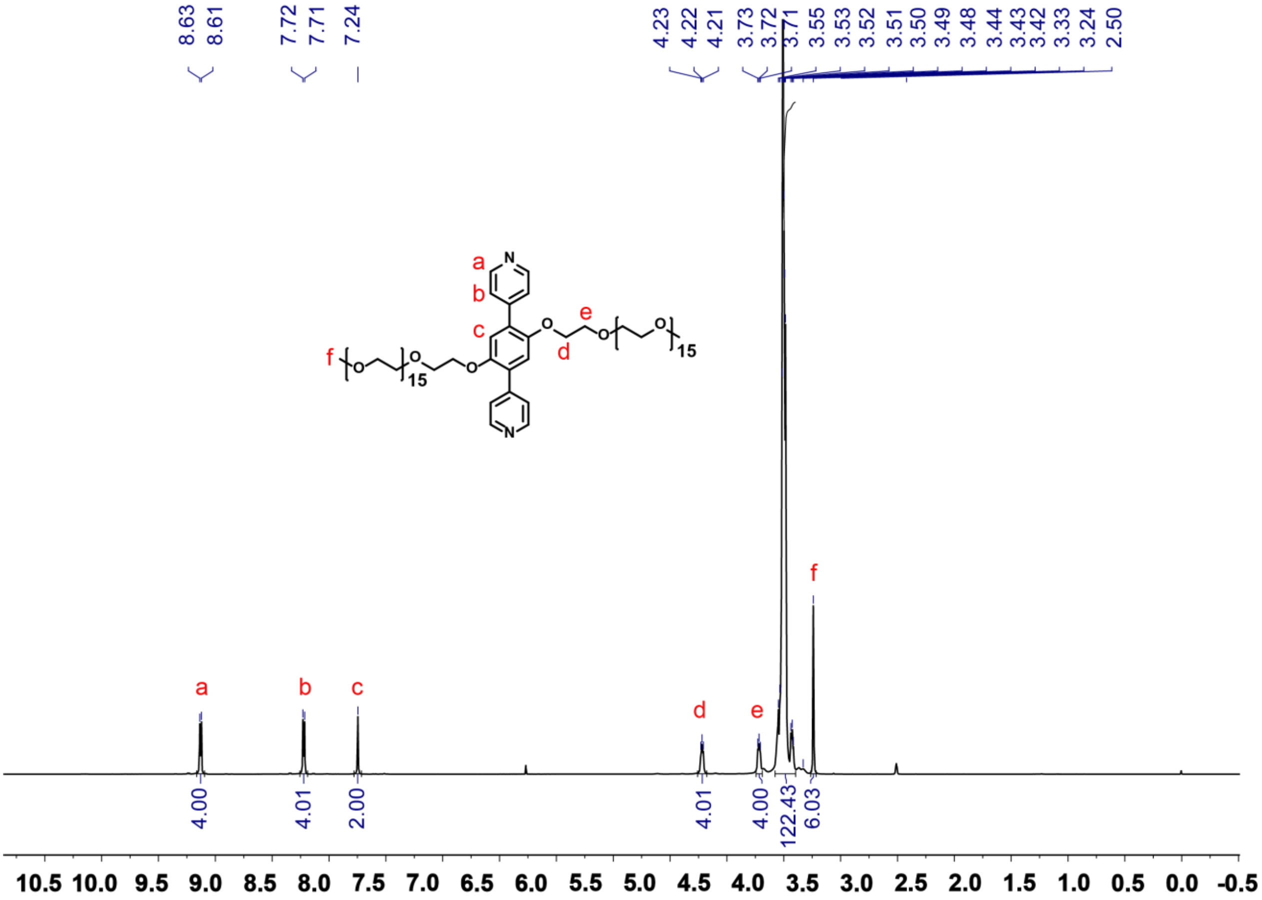


Figure S5. ^{1}H NMR spectrum (400 MHz, DMSO-d_6, 295 K) recorded for **2b**.

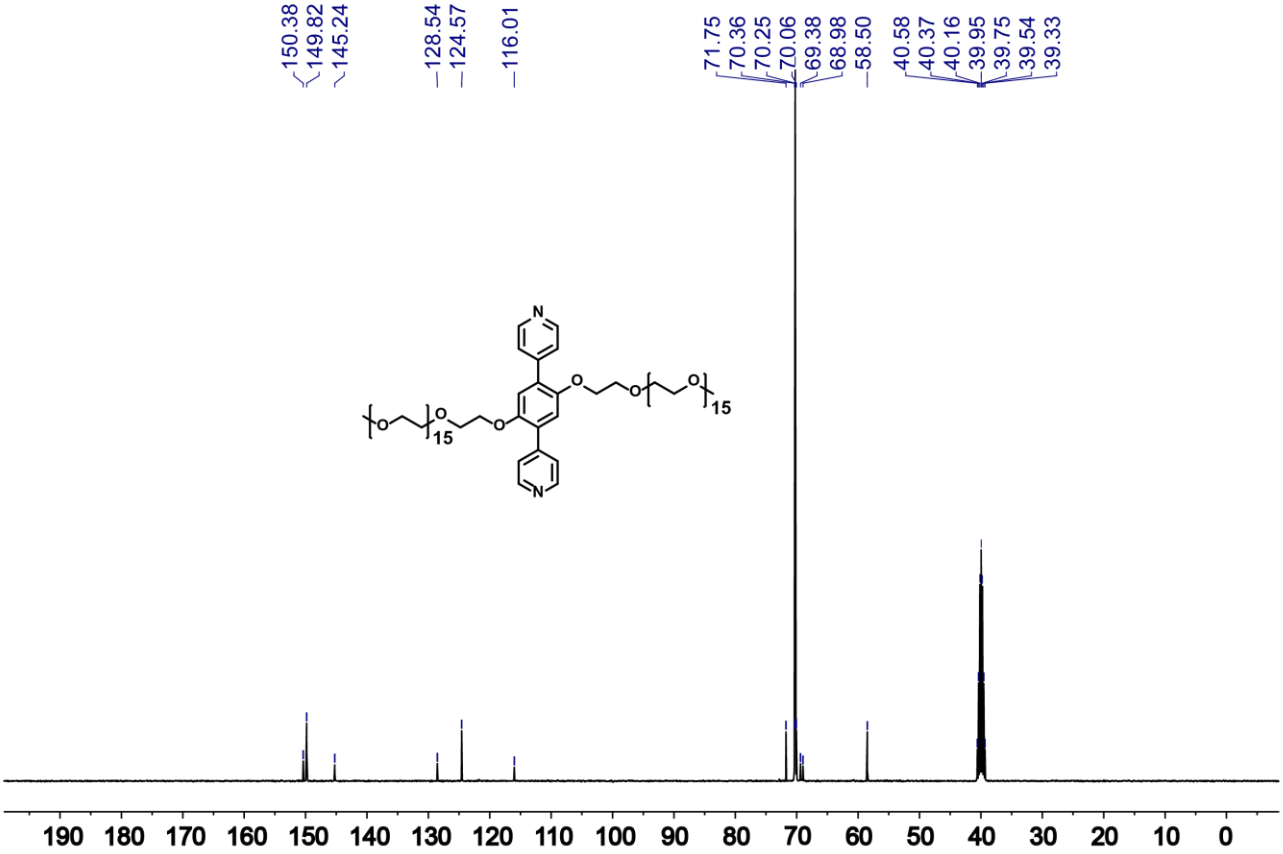
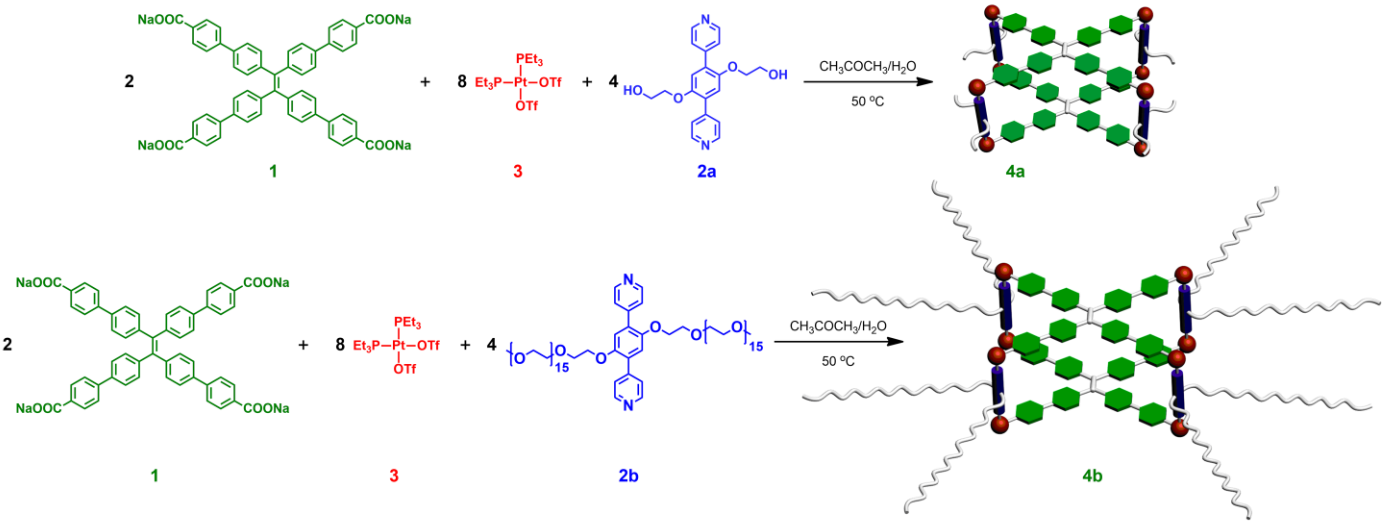


Figure S6. ^{13}C NMR spectrum (100 MHz, DMSO-d_6, 295 K) recorded for **2b**.

*2.4 Synthesis of compound **4a** and **4b***



1 (2.25 mg, 2.50 μmol), **2** (10.0 μmol) and **3** (3.05 mg, 5.00 μmol) were mixed in a 1:4:2 molar ratio and dissolved in acetone/water (1.0 mL, 4:1, v/v). The whole reaction mixture was heated at 50℃ for 12 h and then cooled to room temperature. The solvent was removed by nitrogen flow. The residue was redissolved in acetone (1.0 mL), filtered and the filtrate was poured into ethyl ether (10.0 mL) to give a precipitate, which was collected by centrifugation to give cage **4a** (8.82 mg, 92%) as a pale yellow solid or **4b** (14.51 mg, 88%) as a darl green oil. **4a**: ^{1}H NMR (600 MHz, CD_3COCD_3, 295 K): 8.82–8.73 (m, 16H), 7.92 (d, J = 5.9 Hz, 16H), 7.59 (d, J = 8.5 Hz, 16H), 7.36 (d, J = 8.5 Hz, 16H), 7.29 (d, J = 8.5 Hz, 16H), 7.13 (s, 8H), 7.00 (d, J = 8.5 Hz, 16H), 3.93–3.86 (m, 16H), 3.83 (t, J = 5.6 Hz, 8H), 3.61 (dd, J = 9.7, 5.3 Hz, 16H). ^{13}C NMR (100 MHz, CD_3COCD_3, 295 K): 170.83, 151.18, 151.12, 149.55, 148.60, 143.21, 142.15, 140.73, 138.10, 135.05, 134.99, 131.71, 129.55, 127.74, 126.14, 125.70, 124.12, 123.11, 119.91, 116.18, 71.64, 60.27, 15.54, 15.16, 14.52, 14.14, 7.55, 7.51, 7.46, 7.42. ^{31}P{^{1}H} NMR (121.4 MHz, CD_3COCD_3, 295 K): 5.54 ppm (d, $^2J_{P\text{-}P}$ = 15.8 Hz, ^{195}Pt satellites, $^1J_{Pt\text{-}P}$ = 2507 Hz), 0.32 ppm (d, $^2J_{P\text{-}P}$ = 15.8 Hz, ^{195}Pt satellites, $^1J_{Pt\text{-}P}$ = 2507 Hz). ESI-TOF-MS: m/z 809.5500 [**4a** – 8OTf]$^{8+}$, 1129.1058 [**4a** – 6OTf]$^{6+}$, 1385.1743 [**4a** – 5OTf]$^{5+}$, 1768.7821 [**4a** – 4OTf]$^{4+}$, 2407.7954 [**4a** – 3OTf]$^{3+}$. **4b**: ^{1}H NMR (400 MHz, CD_3COCD_3, 295 K): 8.93 (d, J = 6.2 Hz, 16H), 8.08 (d, J = 6.2 Hz, 16H), 7.76 (d, J = 8.4 Hz, 17H), 7.55 (d, J = 8.4 Hz, 16H), 7.48 (d, J = 8.4 Hz, 16H), 7.29 (s, 8H), 7.18 (d, J = 8.4 Hz, 16H), 4.08 (s, 16H), 3.70–3.47 (m, 657H), 3.34–3.28 (m, 31H). ^{13}C NMR (100 MHz, CD_3COCD_3, 295 K): 170.67, 151.17, 150.91, 148.71, 143.29, 142.08, 140.72, 138.07, 135.14, 131.78, 129.62, 127.85, 126.41, 126.21, 125.75, 123.08, 119.88, 116.42, 71.52, 70.38, 70.33, 70.25, 70.18, 70.11, 70.07, 69.96, 69.89, 69.81, 69.75, 69.71, 69.63, 69.21, 57.99, 15.56, 15.17, 14.56, 14.18, 7.59, 7.55, 7.52, 7.49. ^{31}P{^{1}H} NMR (121.4 MHz, CD_3COCD_3, 295 K): 5.45 ppm (d, $^2J_{P\text{-}P}$ = 15.8 Hz, ^{195}Pt satellites, $^1J_{Pt\text{-}P}$ = 2507 Hz), 0.31 ppm (d, $^2J_{P\text{-}P}$ = 15.8 Hz, ^{195}Pt satellites, $^1J_{Pt\text{-}P}$ = 2507 Hz).

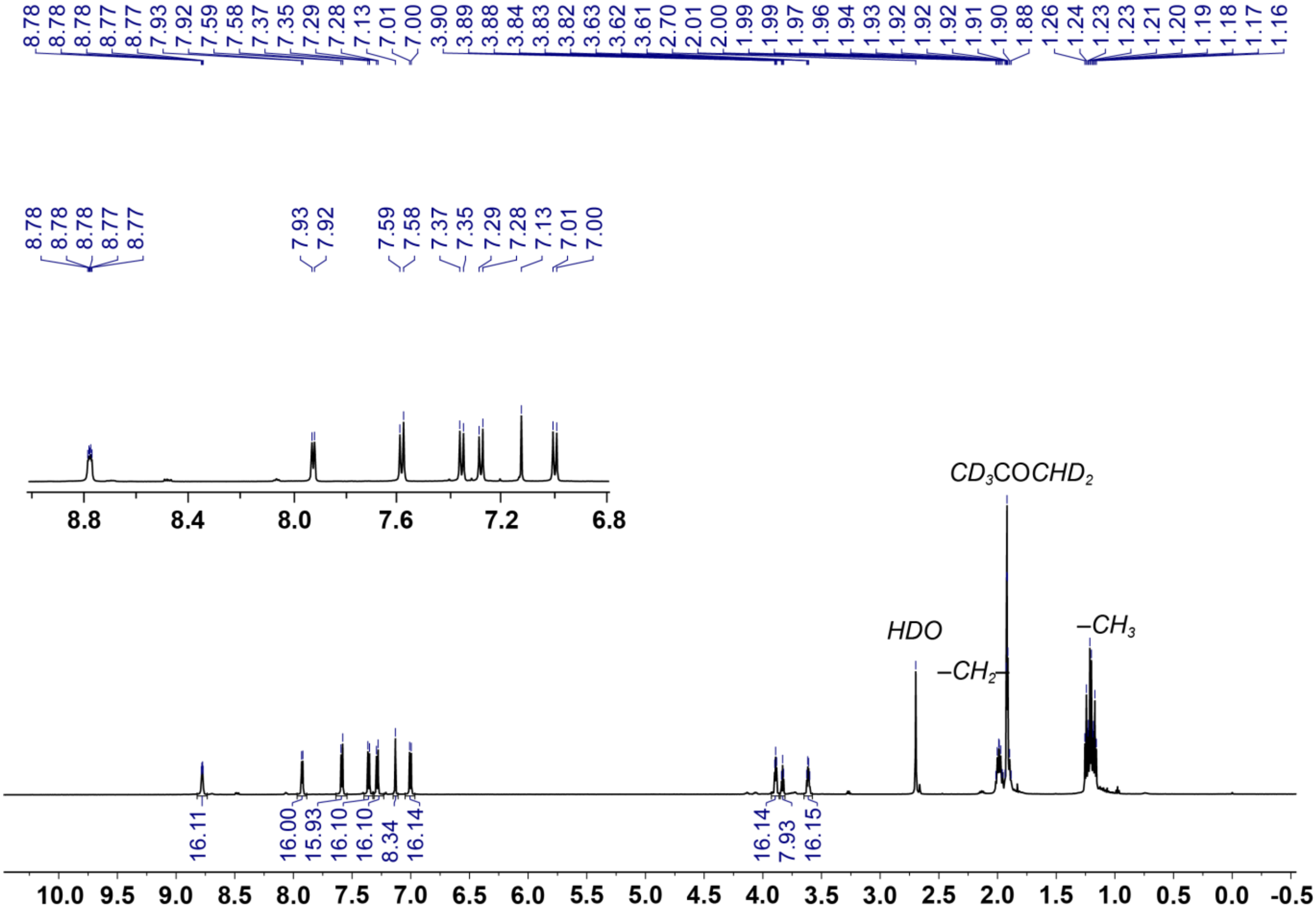


Figure S7. ^{1}H NMR spectrum (400 MHz, CD_3COCD_3, 295 K) recorded for **4a**.

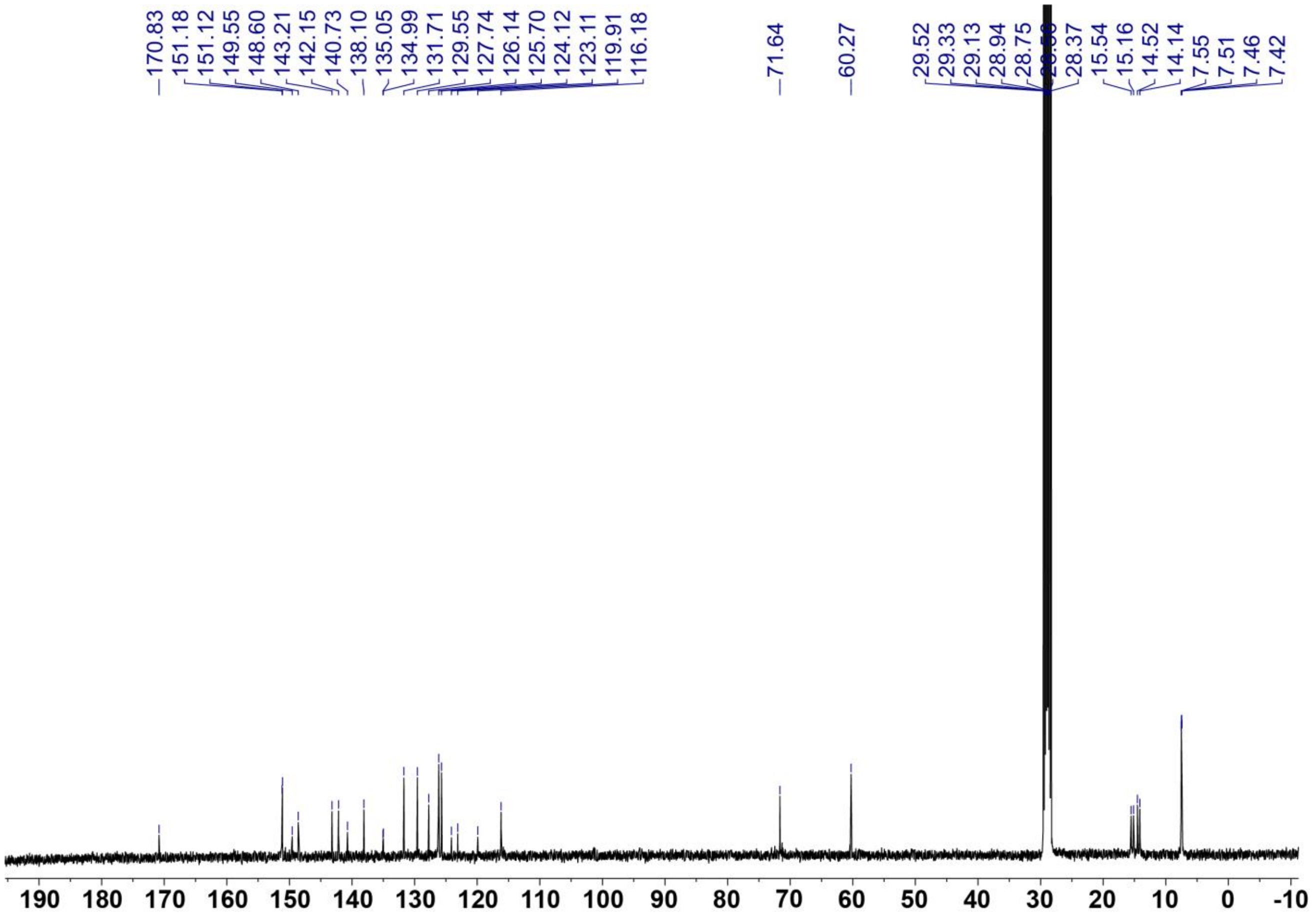


Figure S8. ^{13}C NMR spectrum (100 MHz, CD_3COCD_3, 295 K) recorded for **4a**.

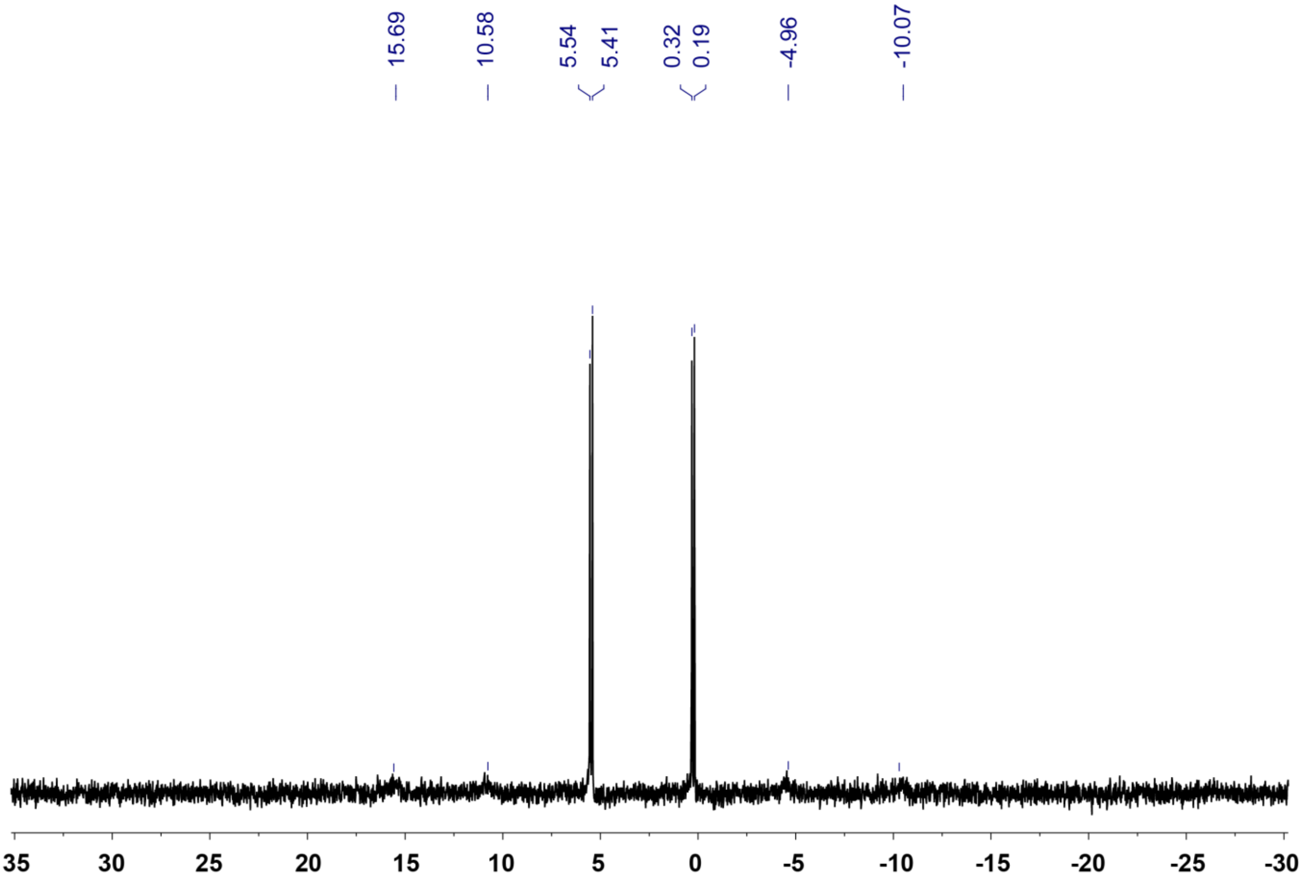


Figure S9. $^{31}P\{^{1}H\}$ NMR spectrum (121.4 MHz, CD_3COCD_3, 295 K) recorded for **4a**.

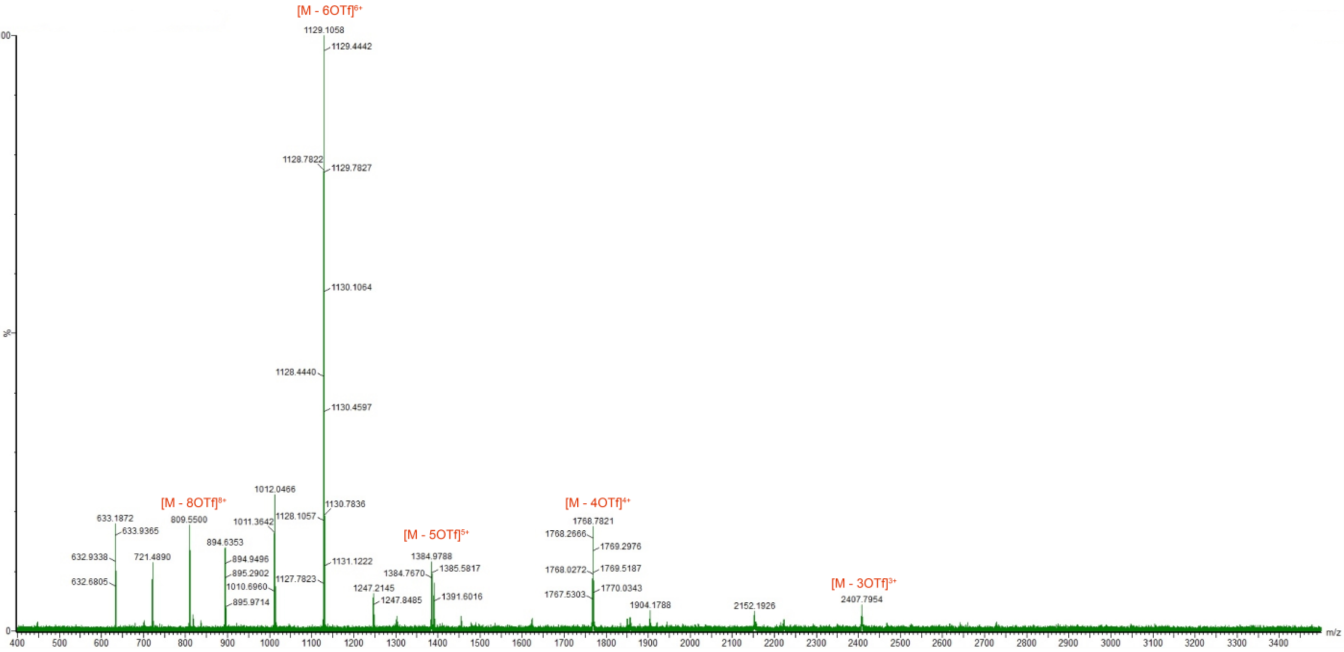


Figure S10. ESI-TOF-MS spectrum of cage **4a**.

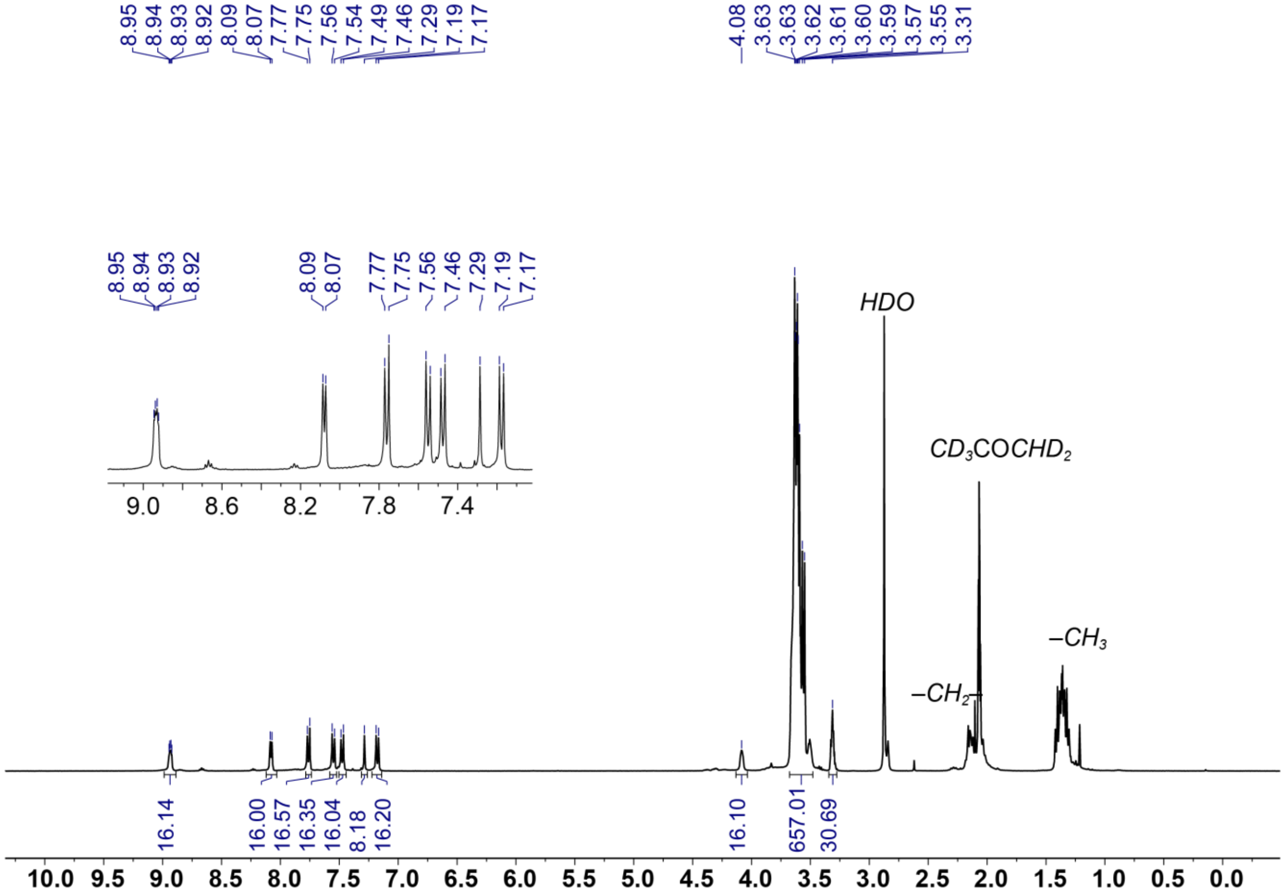


Figure S11. ^{1}H NMR spectrum (400 MHz, CD_3COCD_3, 295 K) recorded for **4b**.

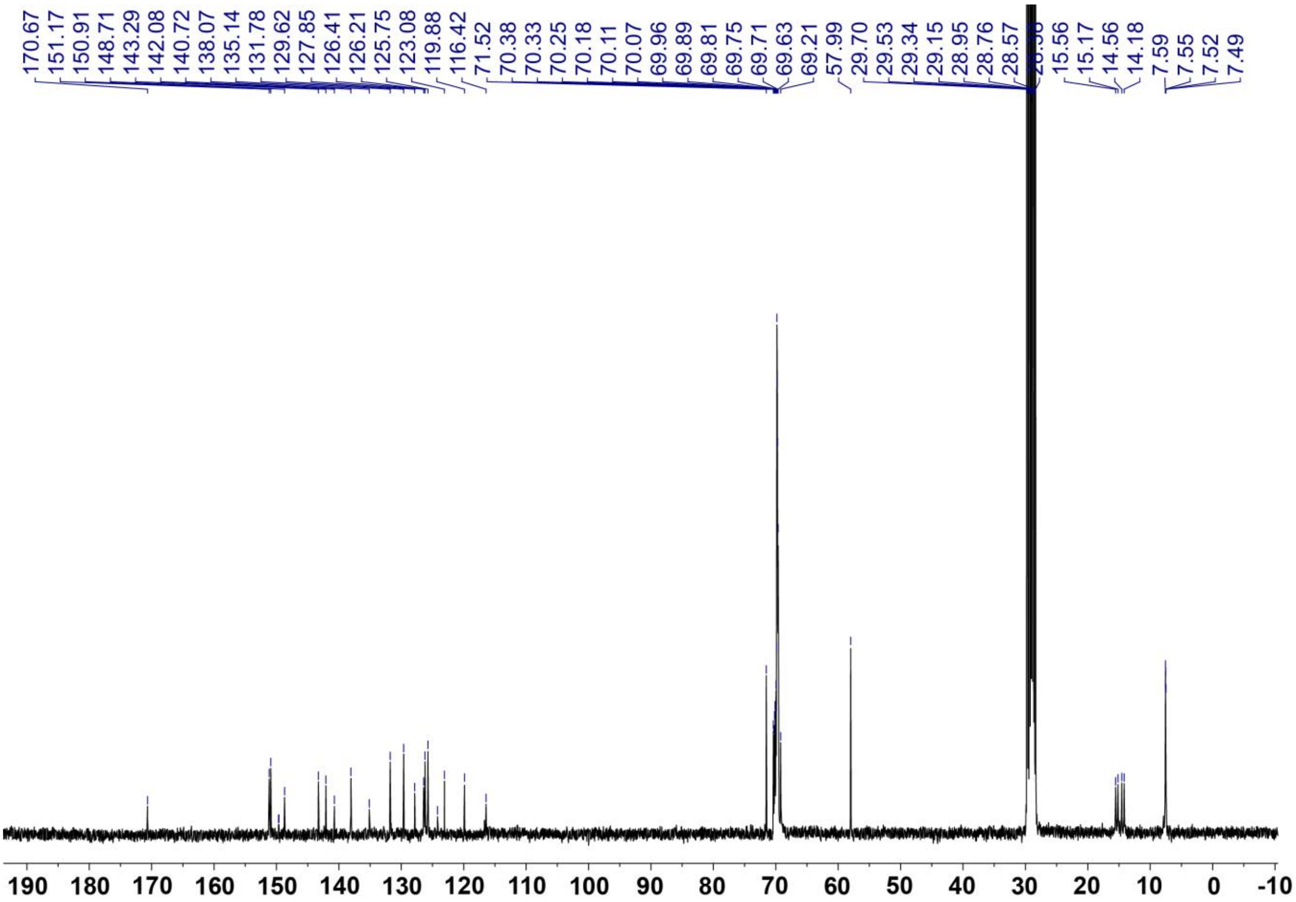


Figure S12. ^{13}C NMR spectrum (100 MHz, CD_3COCD_3, 295 K) recorded for **4a**.

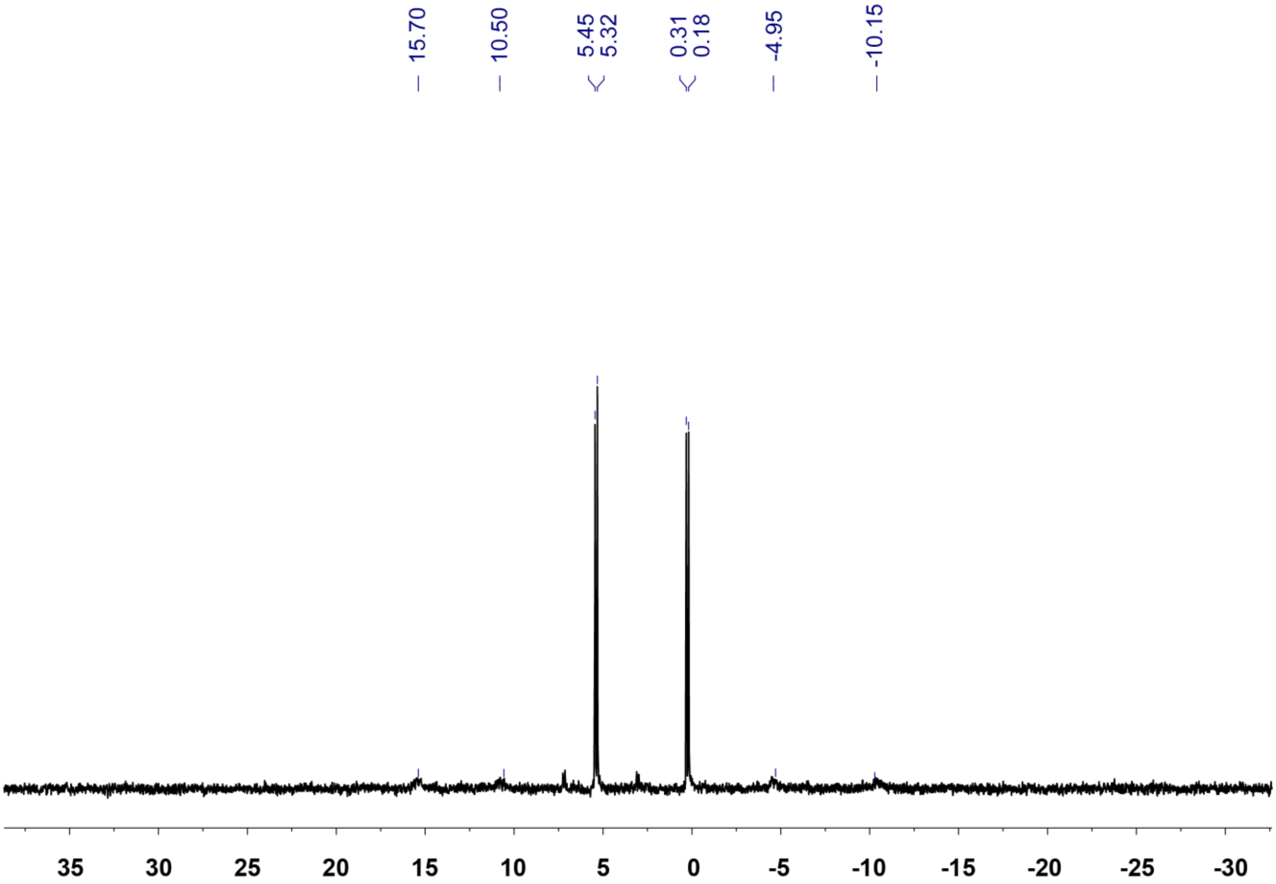


Figure S13. $^{31}P\{^1H\}$ NMR spectrum (121.4 MHz, CD_3COCD_3, 295 K) recorded for **4b**.

3. UV/vis absorption and fluorescence *spectra of **4a** and **4b***

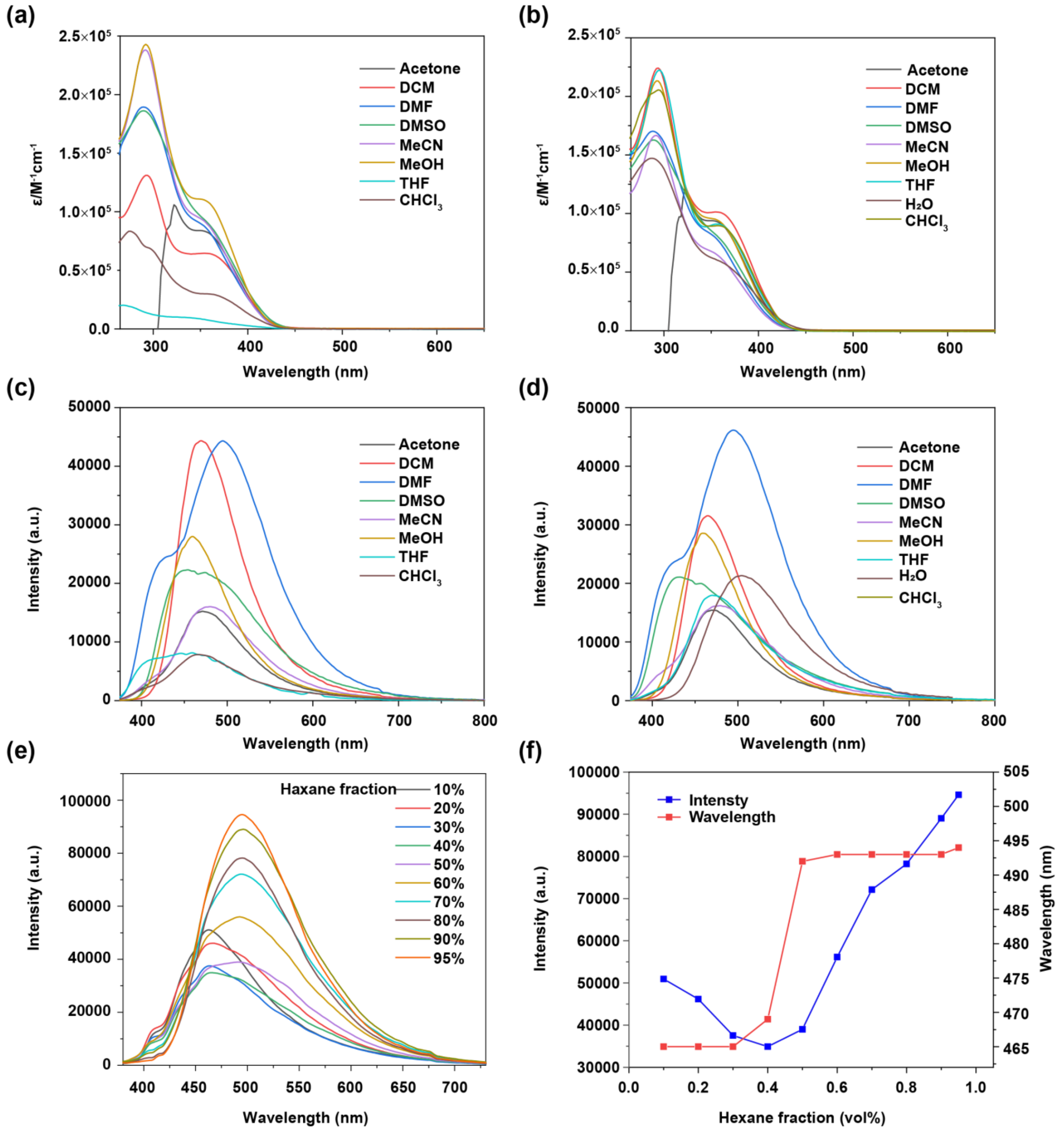
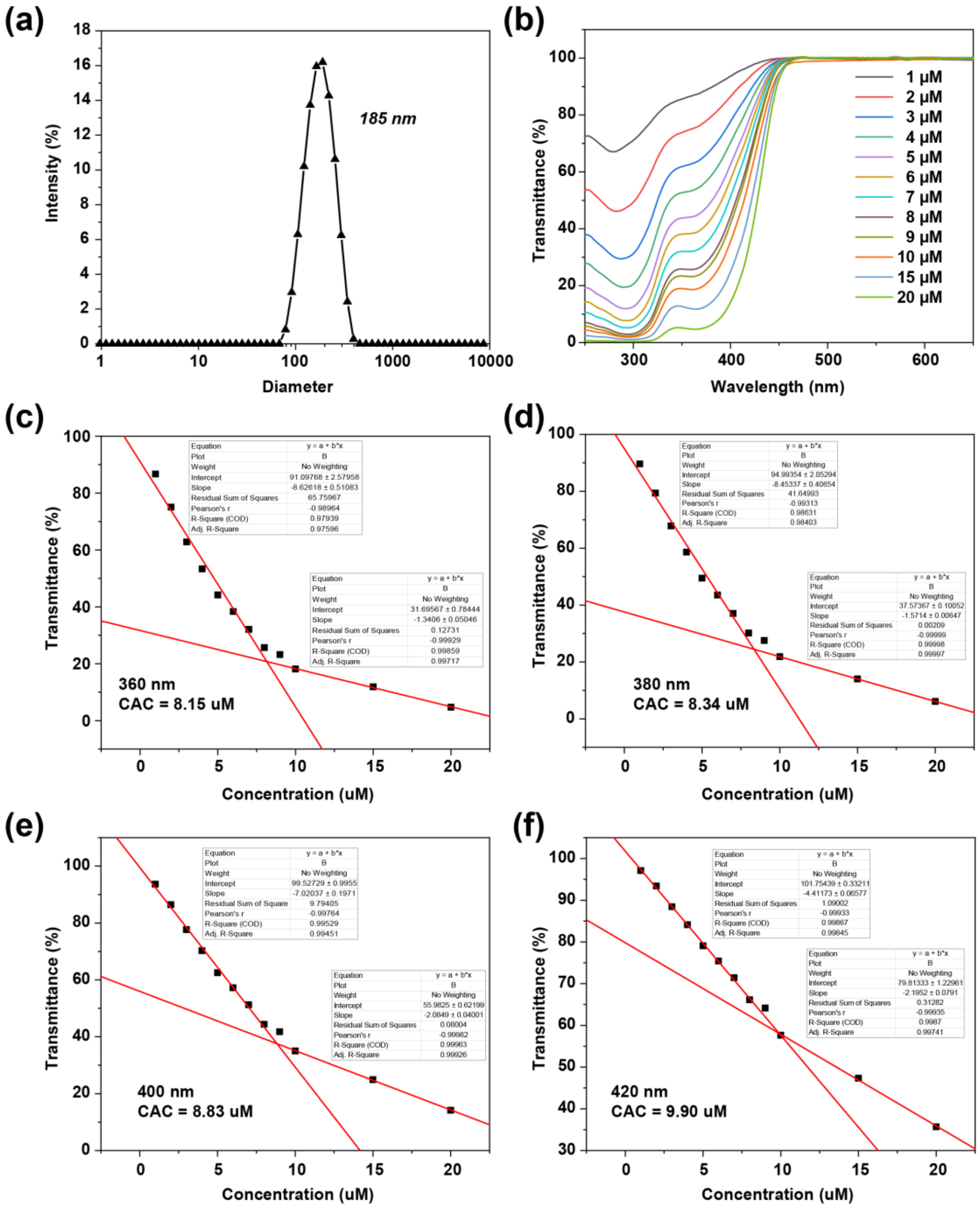


Figure S14. (a, b) UV/vis absorption and (c, d) fluorescence spectra of **4a** (a, c) and **4b** (b, d) in different solvents (λ_{ex} = 365 nm, c =10.0 μM). (e) Fluorescence spectra and (f) plots of maximum emission intensity and wavelength of **4b** versus hexane fraction in CH_2Cl_2/hexane mixtures (λ_{ex} = 365 nm, c = 10.0 μM).

*4. DLS and CAC data of cage **4b** in water*



Equation	y = a + b*x
Plot	B
Weight	No Weighting
Intercept	91.09768 ± 2.57958
Slope	-8.62618 ± 0.51083
Residual Sum of Squares	65.75967
Pearson's r	-0.98964
R-Square (COD)	0.97939
Adj. R-Square	0.97596

Equation	y = a + b*x
Plot	B
Weight	No Weighting
Intercept	31.69567 ± 0.78444
Slope	-1.3406 ± 0.05046
Residual Sum of Squares	0.12731
Pearson's r	-0.99929
R-Square (COD)	0.99859
Adj. R-Square	0.99717

Equation	y = a + b*x
Plot	B
Weight	No Weighting
Intercept	94.99354 ± 2.05294
Slope	-8.45337 ± 0.40654
Residual Sum of Squares	41.64993
Pearson's r	-0.99313
R-Square (COD)	0.98631
Adj. R-Square	0.98403

Equation	y = a + b*x
Plot	B
Weight	No Weighting
Intercept	37.57367 ± 0.10052
Slope	-1.5714 ± 0.00647
Residual Sum of Squares	0.00209
Pearson's r	-0.99999
R-Square (COD)	0.99998
Adj. R-Square	0.99997

Equation	y = a + b*x
Plot	B
Weight	No Weighting
Intercept	99.52729 ± 0.9955
Slope	-7.02037 ± 0.1971
Residual Sum of Square	9.79405
Pearson's r	-0.99764
R-Square (COD)	0.99529
Adj. R-Square	0.99451

Equation	y = a + b*x
Plot	B
Weight	No Weighting
Intercept	55.9825 ± 0.62199
Slope	-2.0849 ± 0.04001
Residual Sum of Squares	0.08004
Pearson's r	-0.99982
R-Square (COD)	0.99963
Adj. R-Square	0.99926

Equation	y = a + b*x
Plot	B
Weight	No Weighting
Intercept	101.75439 ± 0.33211
Slope	-4.41173 ± 0.06577
Residual Sum of Squares	1.09002
Pearson's r	-0.99933
R-Square (COD)	0.99867
Adj. R-Square	0.99845

Equation	y = a + b*x
Plot	B
Weight	No Weighting
Intercept	79.81333 ± 1.22961
Slope	-2.1952 ± 0.0791
Residual Sum of Squares	0.31282
Pearson's r	-0.99935
R-Square (COD)	0.9987
Adj. R-Square	0.99741

Figure S15. (a) DLS data of cage **4b** in water; (b) optical transmittance of different concentrations of **4b** in water; (c) Dependence of the optical transmittance at 360 nm versus the concentration of **4b** in water; (d) Dependence of the optical transmittance at 380 nm versus the concentration of **4b** in water; (e) Dependence of the optical transmittance at 400 nm versus the concentration of **4b** in water; (f) Dependence of the optical transmittance at 420 nm versus the concentration of **4b** in water.

5. *Fluorescent images of **4b** with difference concentrations of eosin Y*

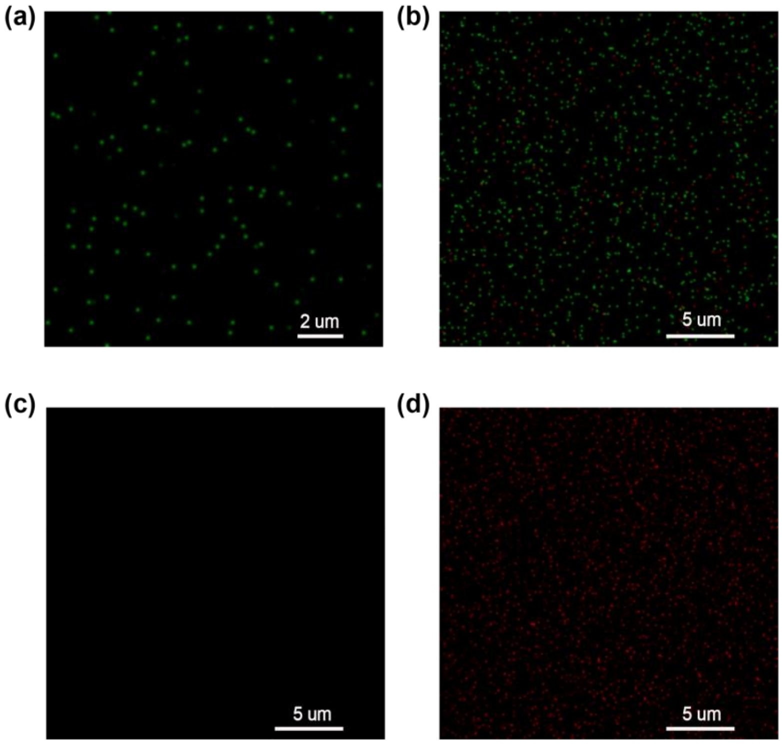


Figure S16. Fluorescent images of (a) metallacage **4b** (c = 10.0 µM) in water (λ_{ex} = 405 nm); (b) metallacage **4b** (c = 10.0 µM) with the addition of eosin Y (c = 5.0 µM) in water (λ_{ex} = 405 nm); (c) eosin Y (c = 5.0 µM) in water (λ_{ex} = 405 nm); (d) eosin Y (c = 5.0 µM) in water (λ_{ex} = 510 nm).

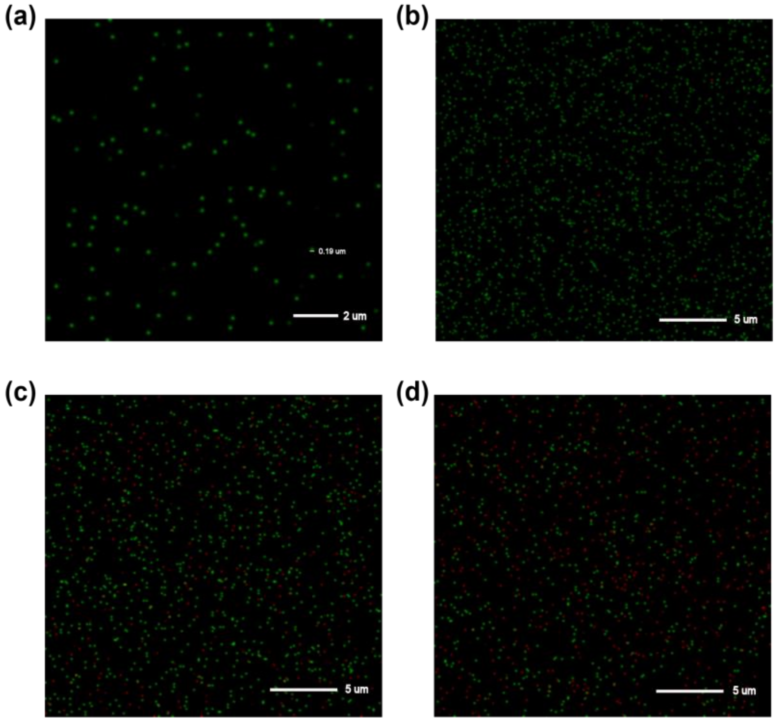


Figure S17. Fluorescent images of (a) **4b** (c = 10.0 µM) in water (λ_{ex} = 405 nm); (b) **4b** (c = 10.0 µM) with the addition of eosin Y (c = 0.2 µM) in water (λ_{ex} = 405 nm); (c) **4b** (c = 10.0 µM) with the addition of eosin Y (c = 5.0 µM) in water (λ_{ex} = 405 nm); (d) **4b** (c = 10.0 µM) with the addition of eosin Y (c = 10.0 µM) in water (λ_{ex} = 405 nm).

6. Antenna effect of the light-harvesting system

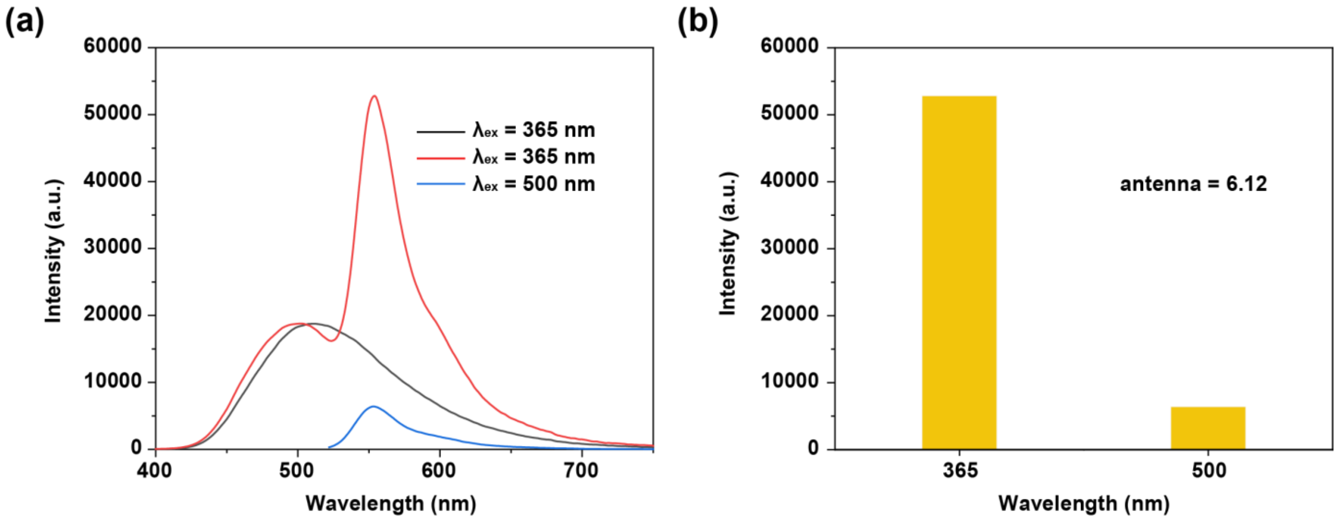
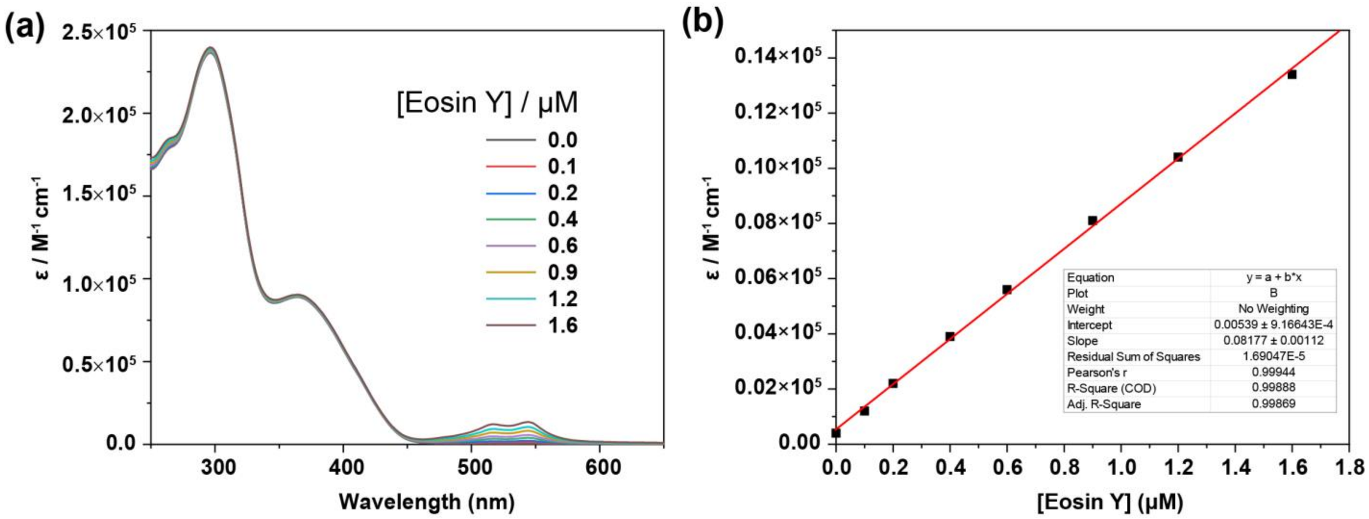


Figure S18. (a) Fluorescence spectra of **4b**/eosin Y (30/1.6 μM) in water, red trace (donor emission, λ_{ex} = 365 nm), blue trace (acceptor emission, λ_{ex} = 500 nm). The black trace represents the fluorescence spectrum (λ_{ex} = 365 nm) of **4b** (30 μM), which was normalized according to the fluorescence intensity at 500 nm of the black trace; (b) The histogram of the eosin Y emission intensities at 554 nm (**4b/** eosin Y = 30/1.6 μM) in water on excitation of the donor (λ_{ex} = 365 nm) and acceptor (λ_{ex} = 500 nm).

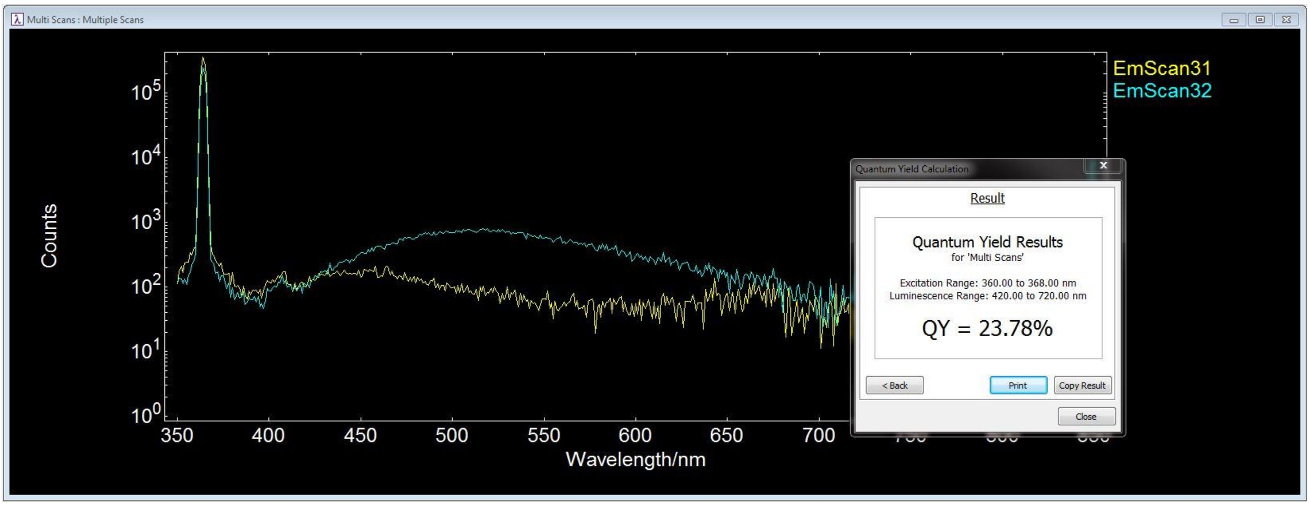
7. UV/vis absorption of the light-harvesting system



Equation	y = a + b*x
Plot	B
Weight	No Weighting
Intercept	0.00539 ± 9.16643E-4
Slope	0.08177 ± 0.00112
Residual Sum of Squares	1.69047E-5
Pearson's r	0.99944
R-Square (COD)	0.99888
Adj. R-Square	0.99869

Figure S19. (a) UV/vis absorption of **4b** (c = 30.0 μM) in water with different concentrations of eosin Y. (b) Dependence of the absorption at 546 nm versus the concentration of eosin Y in water.

*8. Absolute fluorescence quantum yield of cage **4b** in water.*



Result

Quantum Yield Results
for 'Multi Scans'

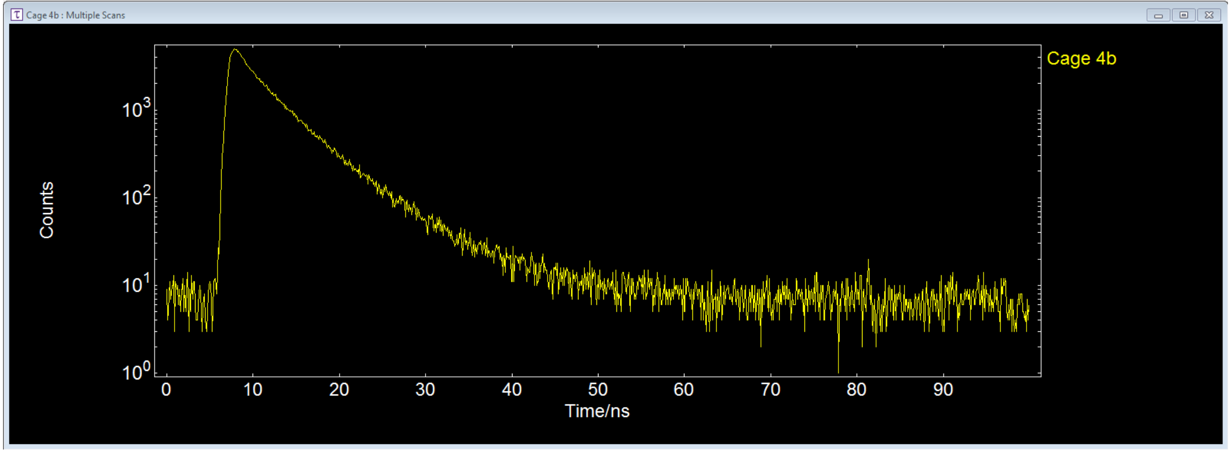
Excitation Range: 360.00 to 368.00 nm
Luminescence Range: 420.00 to 720.00 nm

QY = 23.78%

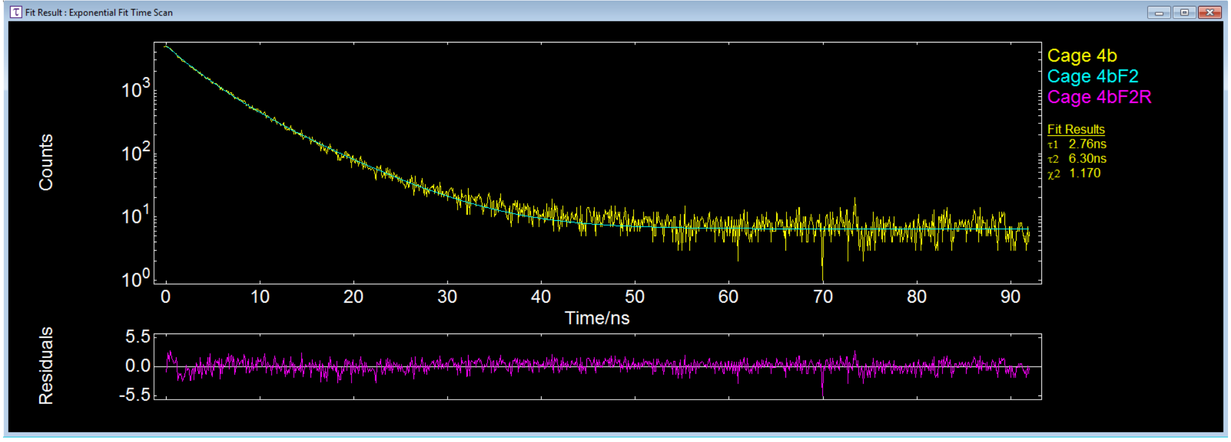
Figure S20. Absolute fluorescence quantum yield of cage **4b** in water.

9. *Fluorescence decay traces and fluorescence lifetime of* ***4b***

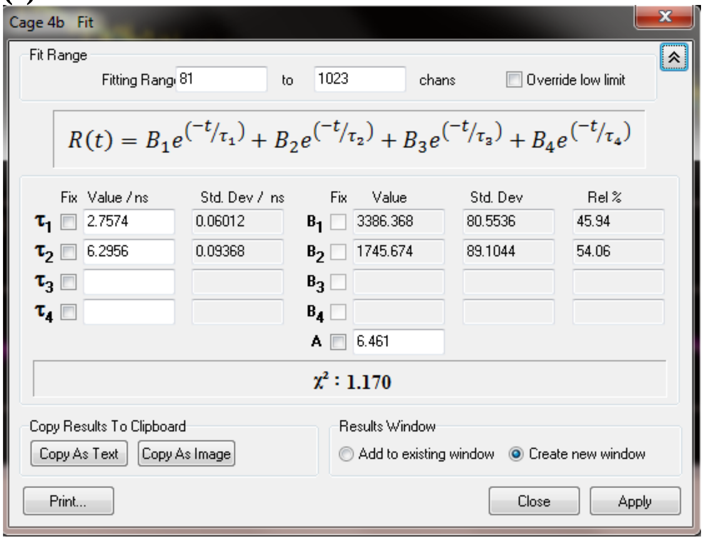
(a)



(b)



(c)



Cage 4b Fit

Fit Range

Fitting Range 81 to 1023 chans ☐ Override low limit

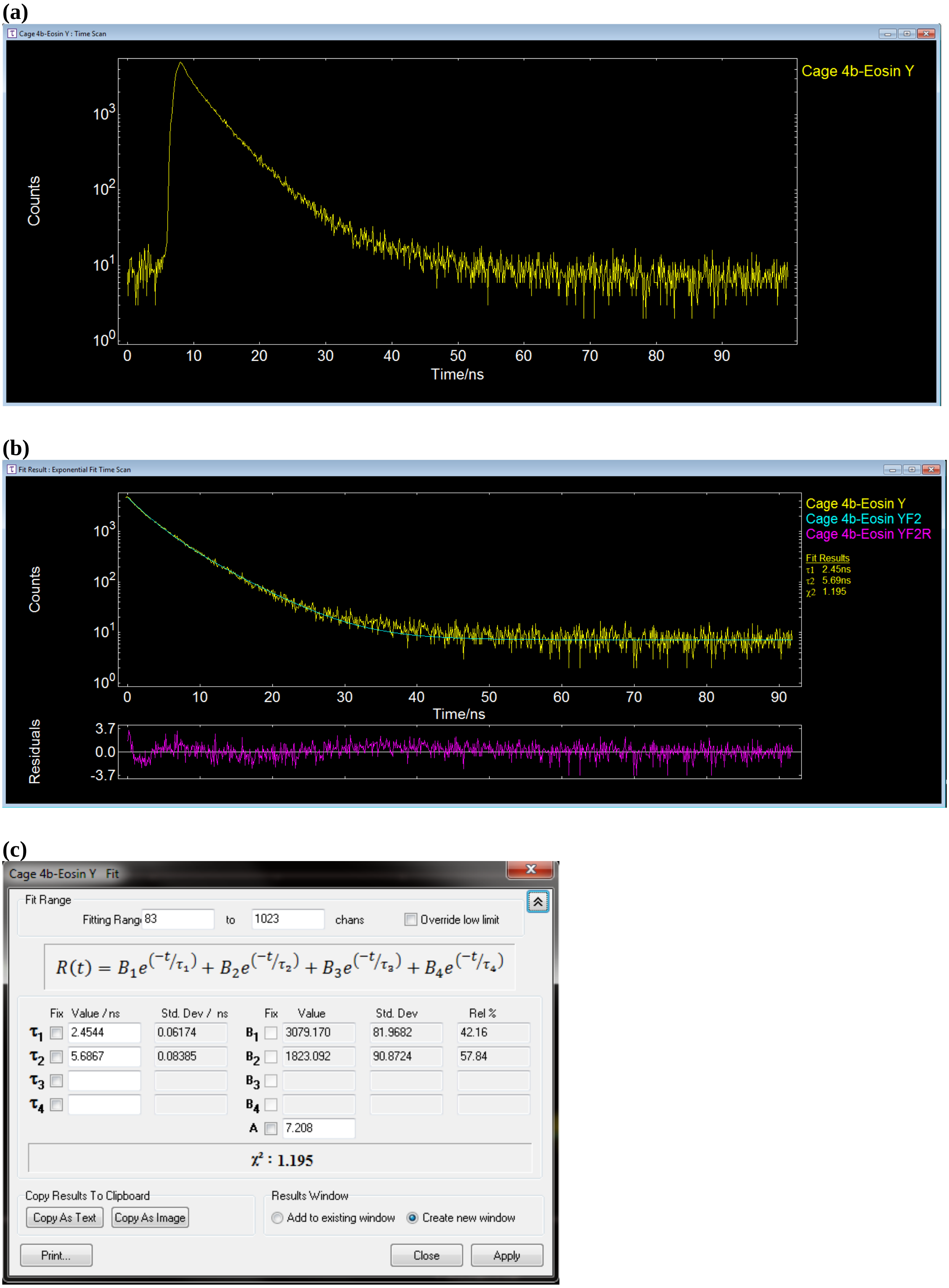
$$R(t) = B_1 e^{(-t/\tau_1)} + B_2 e^{(-t/\tau_2)} + B_3 e^{(-t/\tau_3)} + B_4 e^{(-t/\tau_4)}$$

	Fix	Value / ns	Std. Dev / ns		Fix	Value	Std. Dev	Rel %
τ_1	☐	2.7574	0.06012	B_1	☐	3386.368	80.5536	45.94
τ_2	☐	6.2956	0.09368	B_2	☐	1745.674	89.1044	54.06
τ_3	☐			B_3	☐			
τ_4	☐			B_4	☐			
				A	☐	6.461		

χ^2 : 1.170

Figure S21. Fluorescence decay traces (a and b) and fluorescence lifetime (c) of **4b**.

10. Fluorescence decay traces and fluorescence lifetime of the light-harvesting system



Fitting Rang: 83 to 1023 chans ☐ Override low limit

$$R(t) = B_1 e^{(-t/\tau_1)} + B_2 e^{(-t/\tau_2)} + B_3 e^{(-t/\tau_3)} + B_4 e^{(-t/\tau_4)}$$

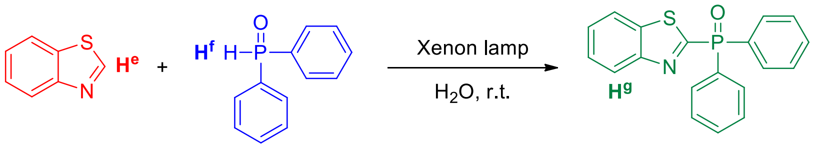
	Fix	Value / ns	Std. Dev / ns		Fix	Value	Std. Dev	Rel %
τ_1	☐	2.4544	0.06174	B_1	☐	3079.170	81.9682	42.16
τ_2	☐	5.6867	0.08385	B_2	☐	1823.092	90.8724	57.84
τ_3	☐			B_3	☐			
τ_4	☐			B_4	☐			
				A	☐	7.208		

χ^2 : 1.195

○ Add to existing window ● Create new window

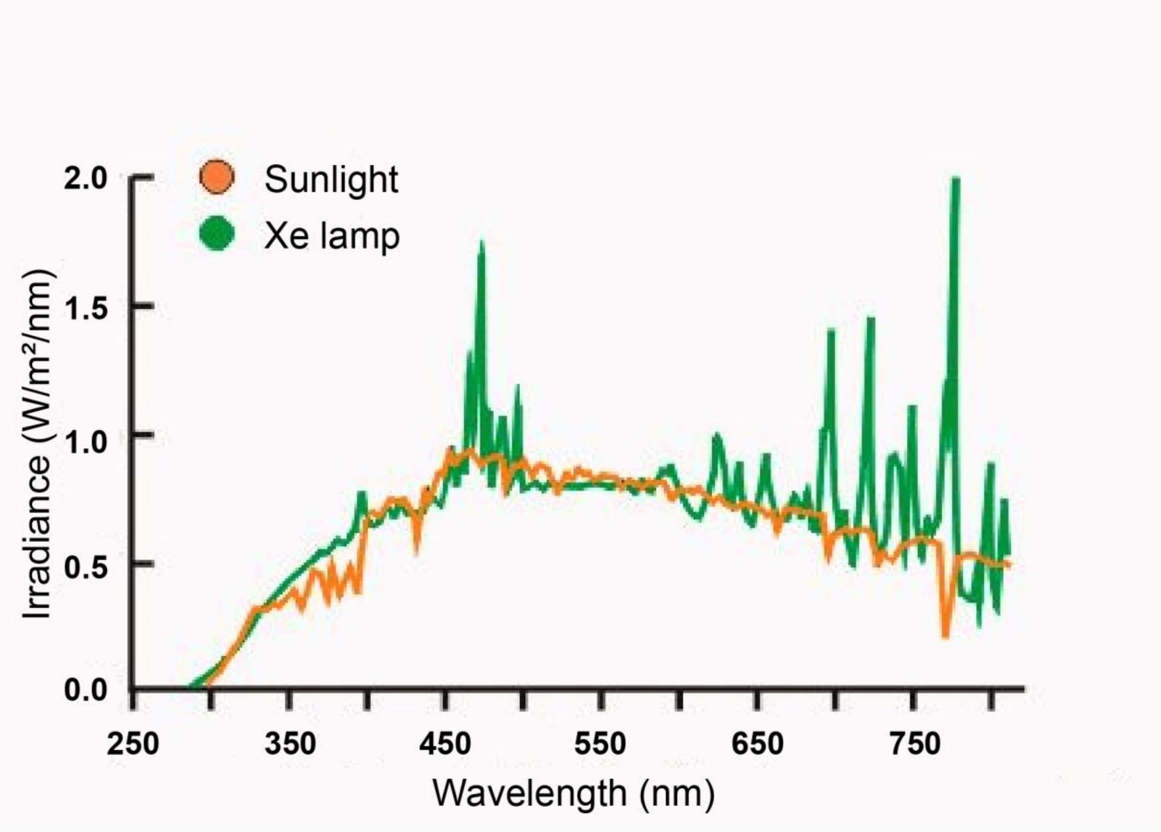
Figure S22. Fluorescence decay traces (a and b) and fluorescence lifetime (c) of light-harvesting system based on **4b** and eosin Y.

11. Study of the catalytic activities of free eosin Y and the light-harvesting system



5 **6** **7**

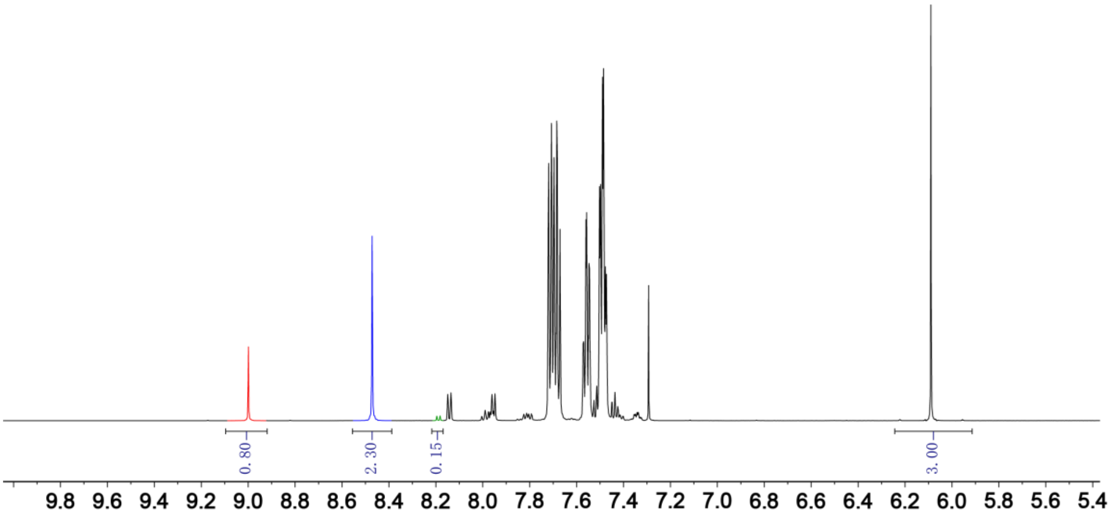
Benzothiazole **5** (50 μmol, 1 equiv.), diphenylphosphine oxide **6** (150 μmol, 3 equiv.), eosin Y (5 μmol, 5 mol%) or cage **4b**/esoin Y (5 μmol, 5 mol%) were dissolved in water (1.5 mL) to a 25 mL Schlenk tube. The mixture was cooled by liquid nitrogen, degassed and purged with nitrogen for six times, and then stirred and irradiated by Xenon lamp (150 mW/cm^2) at room temperature for corresponding time. After that, 20 mL ethyl acetate was added to the resulting mixture. The organic layer was collected and concentrated after washed with saturated $NaHCO_3$ solution and brine. The solvents removed *in vacuo* give light orange oil. The residue was collected and dissolved in deuterated chloroform (0.8 mL) using 1,3,5-trimethoxybenzene (62.5 mM) as the reference to calculate the conversions and yields of the reaction based on the integrals.



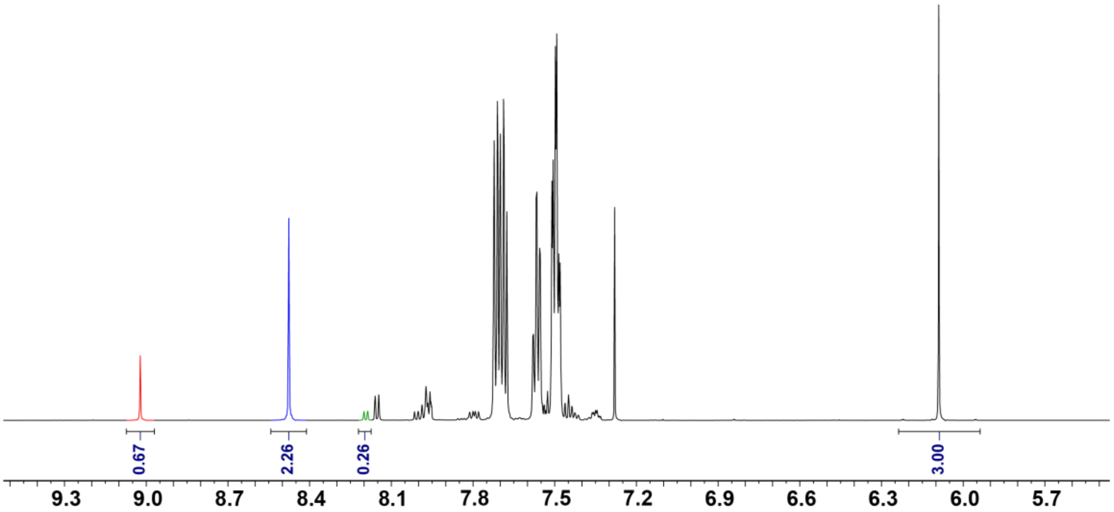
Figrue S23. The spectrum of the sunlight and Xe lamp.

Table S1. Optimization of reaction conditions for the synthesis of **7**.

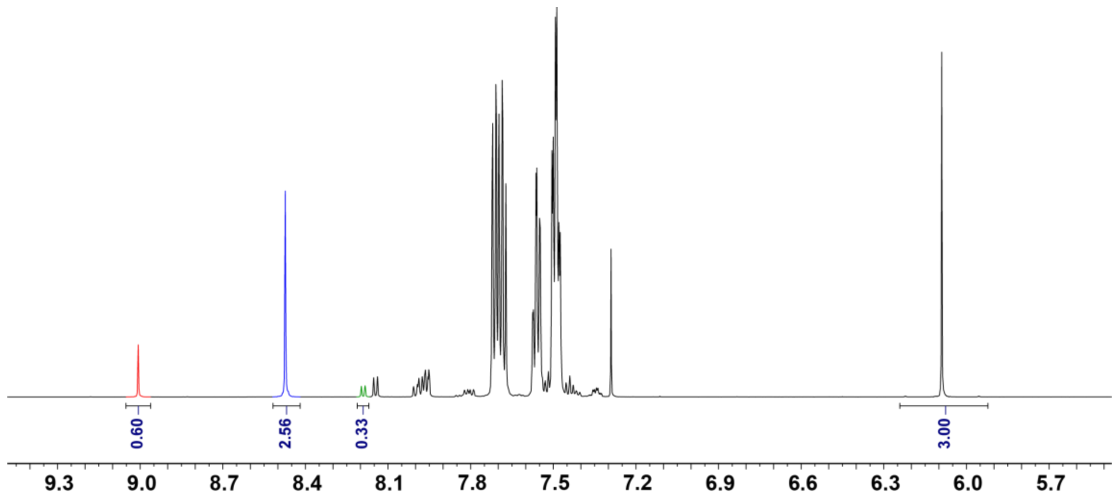
Entry	Additive	Time (h)	Conversion of **5**	Yield of **7**
1	eosin Y	4	20%	15%
2	eosin Y	8	33%	26%
3	eosin Y	12	40%	33%
4	eosin Y	16	46%	38%
5	eosin Y	24	60%	46%
6	eosin Y + cage **4b**	4	25%	21%
7	eosin Y + cage **4b**	6	45%	37%
8	eosin Y + cage **4b**	8	66%	54%
9	eosin Y + cage **4b**	10	83%	59%
10	eosin Y + cage **4b**	12	88%	65%
11	cage **4b**	12	1%	0%



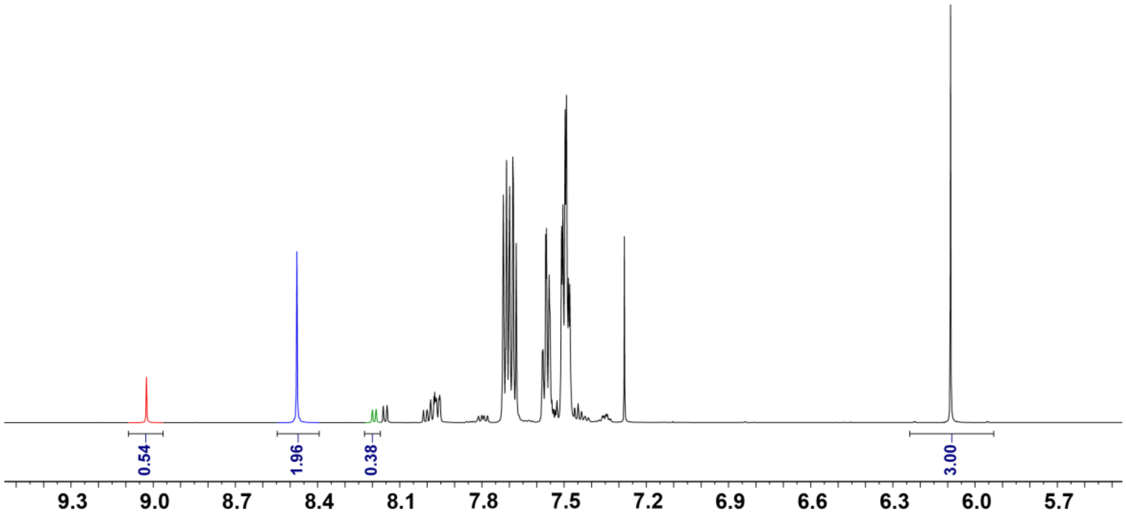
Figrue S24. Partial ^{1}H NMR spectra (600 MHz, CDCl$_3$, 295 K) of the reaction using eosin Y as the photocatalyst at 4 h.



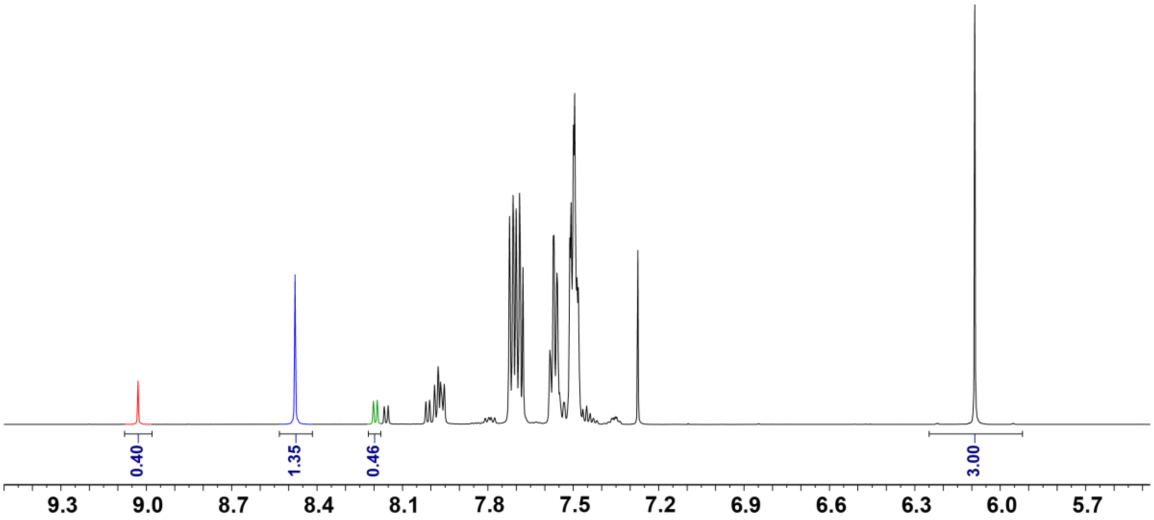
Figrue S25. Partial ^{1}H NMR spectra (600 MHz, CDCl$_3$, 295 K) of the reaction using eosin Y as the photocatalyst at 8 h.



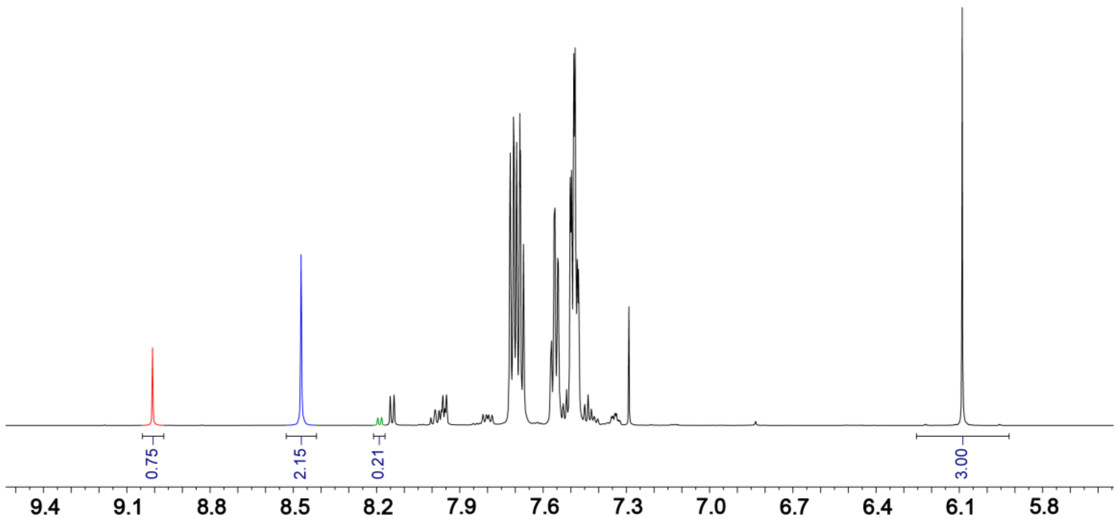
Figrue S26. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 295 K) of the reaction using eosin Y as the photocatalyst at 12 h.



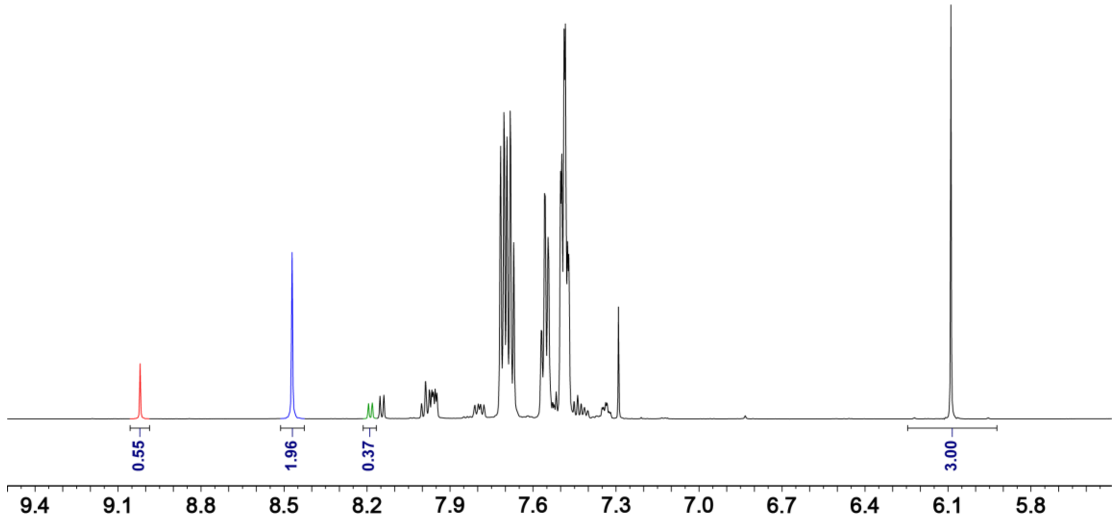
Figrue S27. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 295 K) of the reaction using eosin Y as the photocatalyst at 16 h.



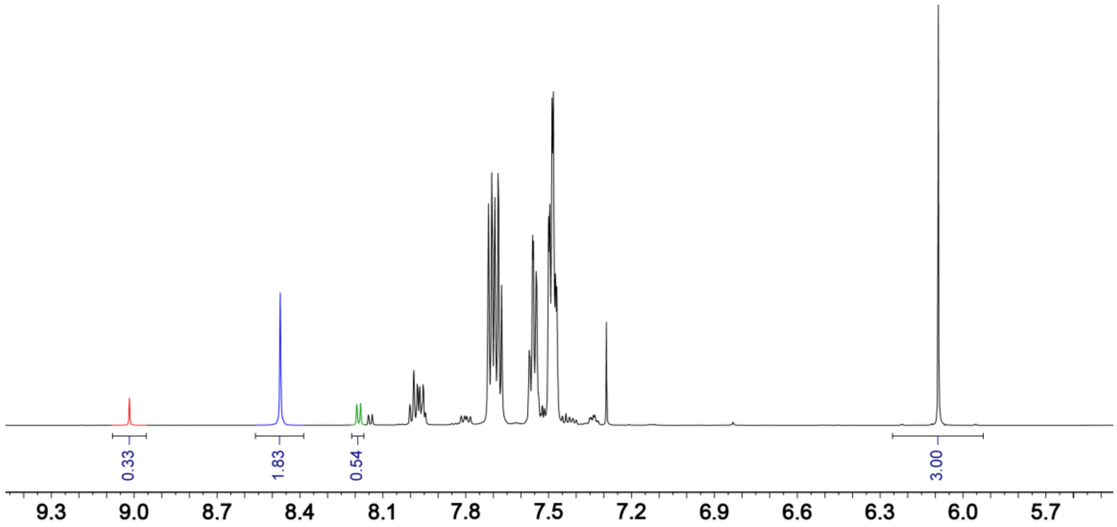
Figrue S28. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 295 K) of the reaction using eosin Y as the photocatalyst at 24 h.



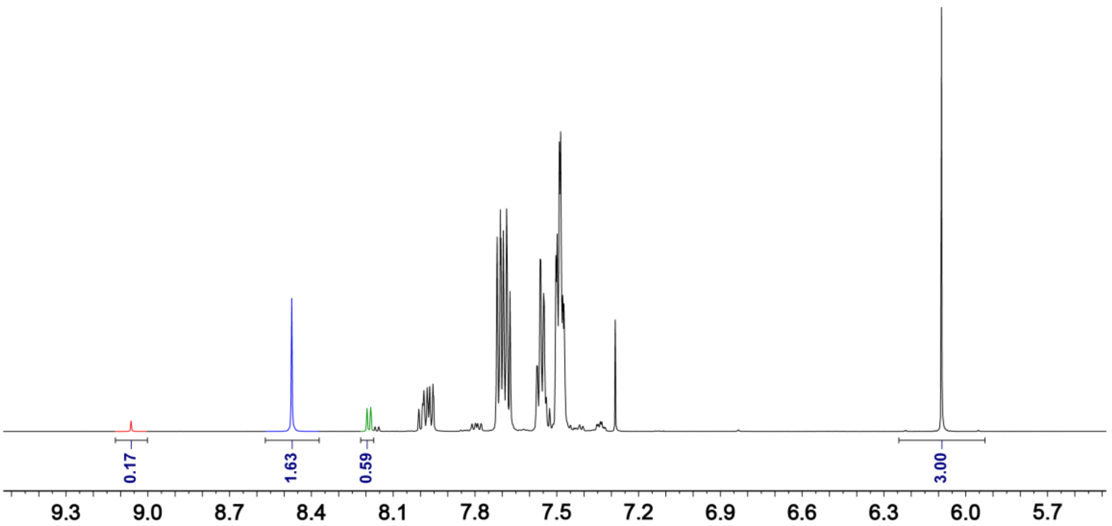
Figrue S29. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 295 K) of the reaction using eosin Y and cage **4b** as the photocatalyst at 4 h.



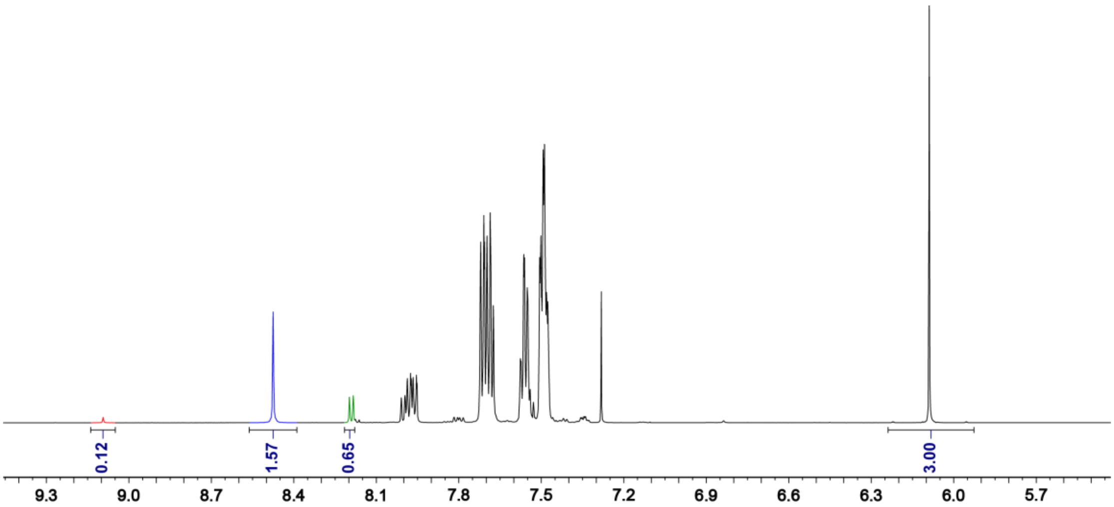
Figrue S30. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 295 K) of the reaction using eosin Y and cage **4b** as photocatalyst at 6 h.



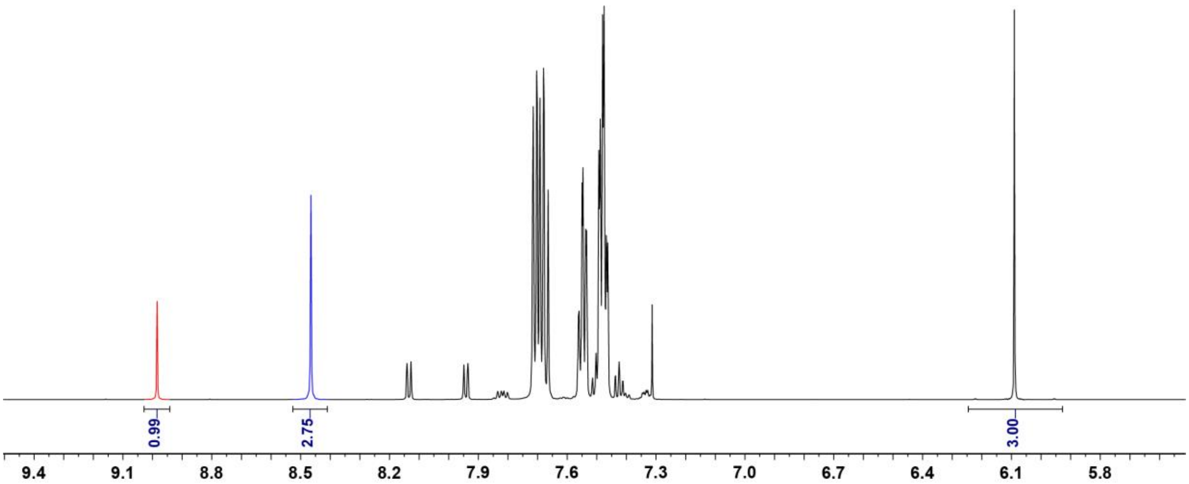
Figrue S31. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 295 K) of the reaction using eosin Y and cage **4b** as photocatalyst at 8 h.



Figrue S32. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 295 K) of the reaction using eosin Y and cage **4b** as photocatalyst at 10 h.



Figrue S33. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 295 K) of the reaction using eosin Y and cage **4b** as photocatalyst at 12 h.



Figrue S34. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 295 K) of the reaction using cage **4b** as photocatalyst at 12 h.

12. Reference

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S2. Y. Zheng, Z. Zhao, M. Wang, K. Ghosh, J. B. Pollock, T. R. Cook, P. J. Stang, *J. Am. Chem. Soc.* **2010**, *132*, 16873-16882.

S3. M. M. Abdellatif, K. Nomura, *Org. Lett.* **2013**, *15*, 1618-1621.