

Supporting Information

1. Synthesis procedures

Synthesis of 2-[2-(*N,N'*-di-*tert*-butoxycarbonylguanidino)ethoxy] ethanol (donated as BocGEE)

GEEMA was prepared according to established methods¹. A solution of *N,N'*-bis(*tert*-butoxycarbonyl)-1*H*-pyrazole-1-carboximidine (4.00 g, 12.89 mmol) was added dropwise to a stirred solution of 2-(2-aminoethoxy) ethanol (1.36 g, 12.89 mmol) and *N,N*-diisopropylethylamine (4.99 g, 38.7 mmol) in DCM (50 mL). The reaction was stirred overnight at room temperature, and then washed with 0.15 N HCl, 0.15 N NaHCO₃, and brine. The organic layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. Silica gel column purification with DCM/EtOAc (2/1, v/v) gave the produce as a colorless oil (4.17 g, 93%). ¹H NMR (CDCl₃, 400 MHz): δ 1.50 (s, 9H), 1.51 (s, 9H), 2.45 (br, s, OH, 1H), 3.62 (m, 4H), 3.67 (m, 2H), 3.76 (m, 2H), 8.83 (br, s, NH, 1H), 11.50 (br, s, NH, 1H).

Synthesis of the monomer 2-[2-(*N,N'*-di-*tert*-butoxycarbonylguanidino)ethoxy] ethyl methacrylate (donated as BocGEEMA)

The synthesis of BocGEEMA was described in our previous work¹. BocGEE (4.17g, 12.00mmol) and TEA (2.5 ml, 18.00 mmol) was dissolved in 25ml anhydrous CH₂Cl₂. Methacryloyl chloride (1.38 g, 13.20 mmol) in anhydrous CH₂Cl₂ was added dropwise to the flask at 0 °C under nitrogen atmosphere and stirred for 6h at room temperature. After termination of the reaction with water, the mixture was washed successively with 0.2 N HCl, 0.2 N NaHCO₃, and brine. The orange organic layer was dried over anhydrous Na₂SO₄ and concentrated under vacuum. The colorless oil product (3.89 g, 78%) was afforded with silica gel using DCM/EtOAc (15/1, v/v) as eluent. ¹H NMR (CDCl₃, 400 MHz): δ 1.49 (s, 9H), 1.51 (s, 9H), 1.96 (s, 3H), 3.64 (m, 2H), 3.70 (m, 2H), 3.74 (t, *J* = 4.80 Hz, 2H), 4.31 (t, *J* = 4.71 Hz, 2H), 5.57 (m, 1H), 6.16 (m, 1H), 8.77 (br, s, NH, 1H), 11.48 (br, s, NH, 1H).

Synthesis of macro-initiator CH₃O-PEO₁₁₄-Br

Macro-initiator CH₃O-PEO₁₁₄-Br was synthesized as followed²: CH₃O-PEO₁₁₄-OH (15.0 g, 3.00 mmol) was dissolved in 150 ml toluene. After azeotropic distillation of 50 ml solvent, the mixture was cooled to 30 °C and added TEA (3.20 g, 30.0 mmol) and 2-bromo-2-methylpropionyl bromide (6.90 g, 30.0 mmol) subsequently. The reaction was stirred for five days. The yellowish powder was

obtained after the precipitation in a ten-fold excess of petroleum ether. Then, the crude product was redissolved in about 100 ml THF and precipitated in a ten-fold excess of petroleum ether. Successively discoloring in water with charcoal, extracting with CH₂Cl₂, drying over anhydrous Na₂SO₄, and precipitating in petroleum ether gave the purified product as a white powder. Gel permeation chromatography analysis results (polystyrene standards): $M_{n, GPC} = 7200$ g/mol and the polydispersity index (PDI) = 1.06. ¹H NMR (CDCl₃, 400 MHz): δ 1.94 (s, 6H; C(CH₃)₂Br), 3.38 (s, 3H; OCH₃), 3.65 (bs, 456H; OCH₂CH₂O of PEO). The degree of esterification was 100% determined by ¹H NMR.

Synthesis of the CH₃O-PEO₁₁₄-*b*-PBocGEEMA₂₃ diblock polymers (donated as PEO₁₁₄-*b*-PBGU₂₃)

The preparation of CH₃O-PEO₁₁₄-*b*-PBocGEEMA₂₃ was an ATRP procedure. Macro-initiator CH₃O-PEO₁₁₄-Br (0.23g, 4.62×10^{-5}), monomer BocGEEMA (0.46 g, 1.11×10^{-3} mol), copper (I) bromide (13.24 mg, 9.23×10^{-5} mol), PMDETA (15.59 mg, 9.23×10^{-5} mol), and dry THF (3 mL) were introduced into a glass tube equipped with a magnetic stirring bar. The mixture was degassed by three freeze-vacuum-thaw cycles and the tube was sealed before initiating the polymerization at 35 °C. Three hours later, the reaction was quenched by cooling down in liquid nitrogen and introducing air. The mixture was then diluted with THF and precipitated in petroleum ether/ethyl ether (2/1, v/v). After dissolving the precipitation in CH₂Cl₂ and washing with EDTA solution twice, the copper catalyst was removed. The organic layer was dried over Na₂SO₄, concentrated under reduced pressure, dissolved in 10 ml THF, and precipitated in petroleum ether/ethyl ether (2/1, v/v). Polymer was afforded as a white power after drying under vacuum. Gel permeation chromatography analysis results (polystyrene standards): $M_{n, GPC} = 13600$ g/mol and the polydispersity index (PDI) = 1.14. ¹H NMR (CDCl₃, 400 MHz): δ (ppm), 0.96 (m, 3H; CH₃), 1.49 (s, 18 H, (CH₃)₃C), 1.85 (m, 2H; CH₂), 3.38 (s, 0.13; OCH₃), 3.65 (bs, 26H; OCH₂CH₂O of both EO and BocGEEMA units), 4.09 (s, 2H, COOCH₂), 8.58 (s, 1H; NH), 11.50 (s, 1H; NH). The number of BocGEEMA units was 23, calculated from its ¹H NMR spectrum.

Synthesis of the CH₃O-PEO₁₁₄-*b*-PGEEMA₂₃ diblock polymers (donated as PEO₁₁₄-*b*-PGU₂₃)

The removing of Boc-group from diblock polymer CH₃O-PEO₁₁₄-*b*-PBocGEEMA₂₃ ($M_{n, GPC} = 13600$ g/mol, PDI = 1.14) was completed using trifluoroacetic acid (5 ml) in CH₂Cl₂ (20 ml) for 6 hours at room temperature. The resulting Boc-deprotected polymer, CH₃O-PEO₁₁₄-*b*-PGEEMA₂₃, was

isolated by rotary evaporation and washed with ethyl ether three times. The completeness of the deprotection was confirmed from the disappearance of the ^1H NMR signal at $\delta = 1.49$ ppm (*tert*-Boc). ^1H NMR (D_2O): δ (ppm), 1.00 (m, 3H; CH_3), 1.85 (m, 2H; CH_2), 3.36 (s, 2H; $\text{CH}_2\text{-N}$), 3.64 (bs, 24H; $\text{OCH}_2\text{CH}_2\text{O}$ of both EO and GEEMA units), 4.13 (s, 2H, COOCH_2).

Reference

1. H. B. Wei, S. M. Du, Y. Liu, H. X. Zhao, C. Y. Chen, Z. B. Li, J. Lin, Y. Zhang, J. Zhang and X. H. Wan, *Chem. Commun.*, 2014, **50**, 1447-1450.
2. M. Lemmers, J. Sprakel, I. K. Voets, J. van der Gucht and M. A. C. Stuart, *Angew. Chem. Int. Ed.*, 2014, **50**, 708-711.

2、 Figure and Tables

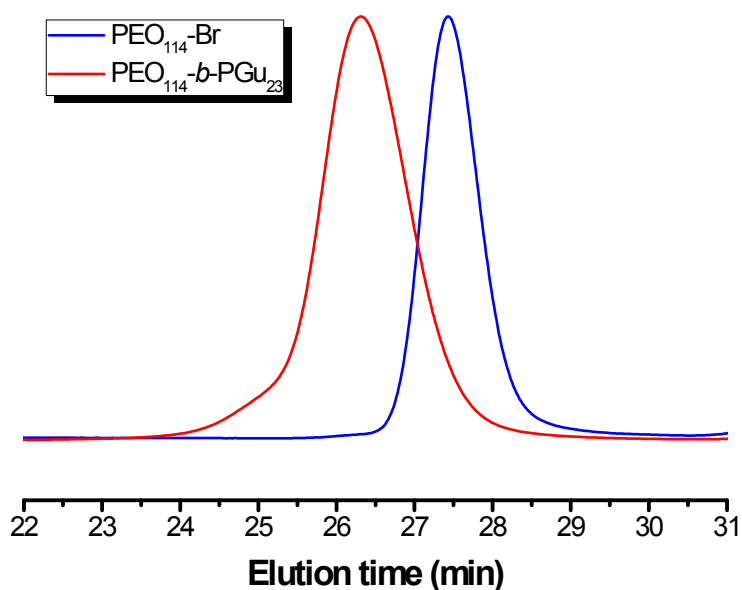


Fig. S1, GPC traces of $\text{PEO}_{114}\text{-Br}$ macroinitiator and $\text{PEO}_{114}\text{-}b\text{-PBG}_{23}$ diblock copolymers.

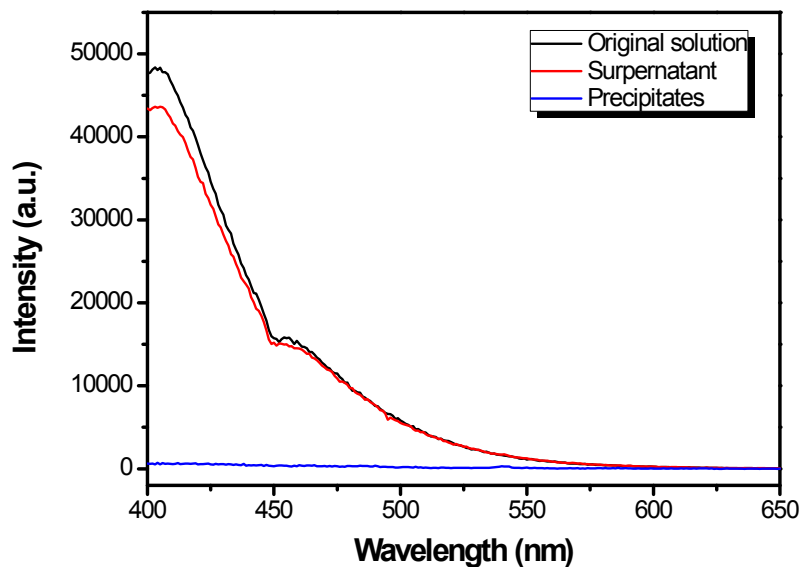


Fig. S2, Emission spectra of the original solution before centrifugation (-), the supernatant solution after centrifugation (-), and the re-dispersed precipitates after centrifugation (-) of TbW₁₀/EuW₁₀/Quinine = 50/1/5 excited 280nm. The concentration of TbW₁₀ is 1.75×10⁻³ M and PEO₁₁₄-*b*-PGu₂₃ is 6.85×10⁻⁴.

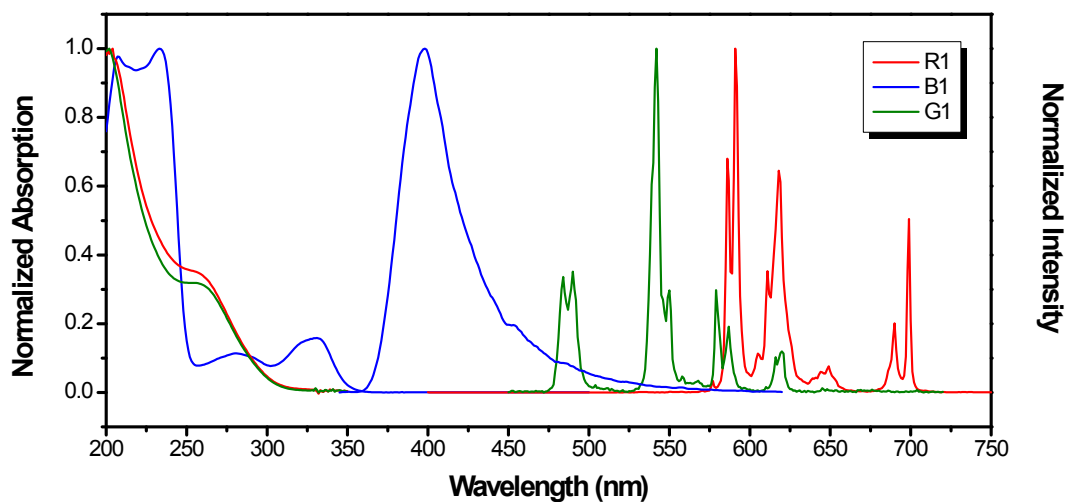


Fig. S3, Absorption and emission spectra of R1, G1, and B1 ($\lambda_{\text{ex}} = 280 \text{ nm}$, $\lambda_{\text{em}} = 400 \text{ nm}$) hybrid solutions. The excitation wavelength is 280 nm.

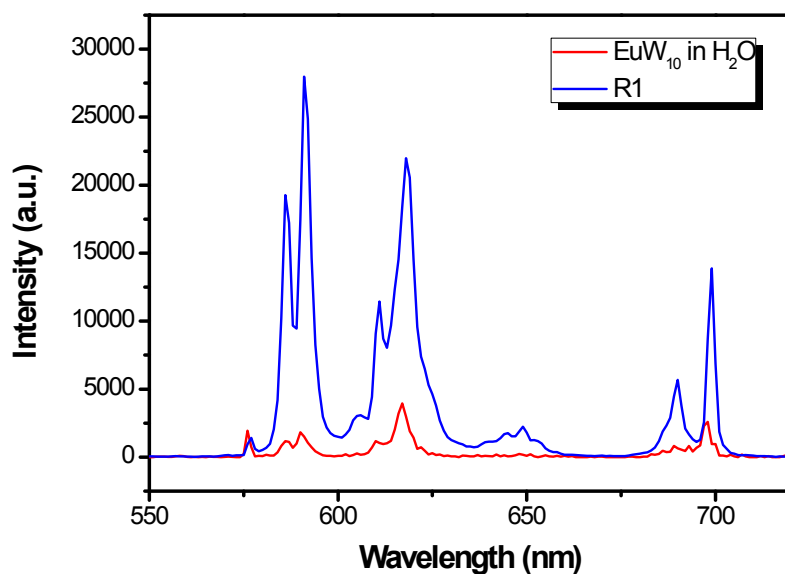


Fig. S4, Emission spectra of EuW_{10} in H_2O (-) and R1 primary solution (-) at an excitation wavelength of 280 nm.

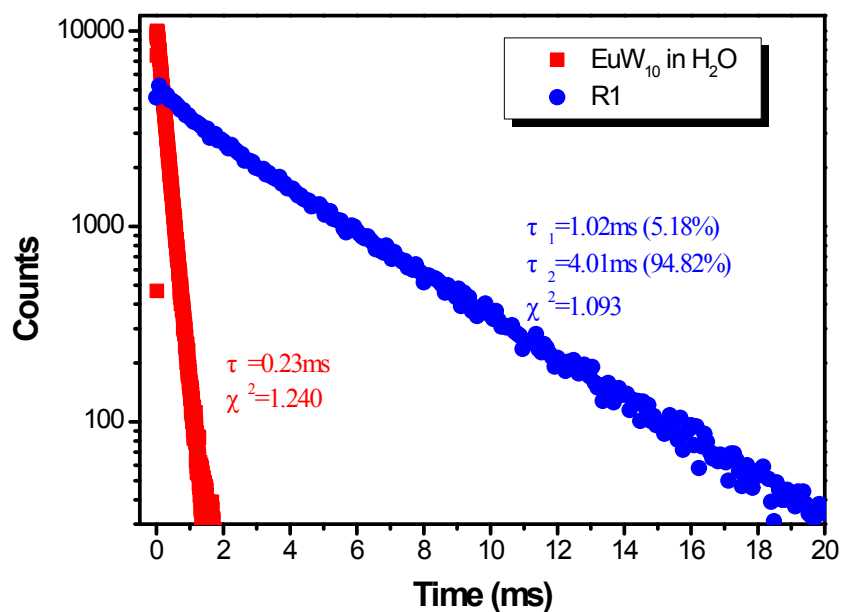


Fig. S5, Emission decay curves of EuW_{10} in H_2O (■) and R1 (●) at an excitation wavelength of 280 nm and monitored at 587 nm.

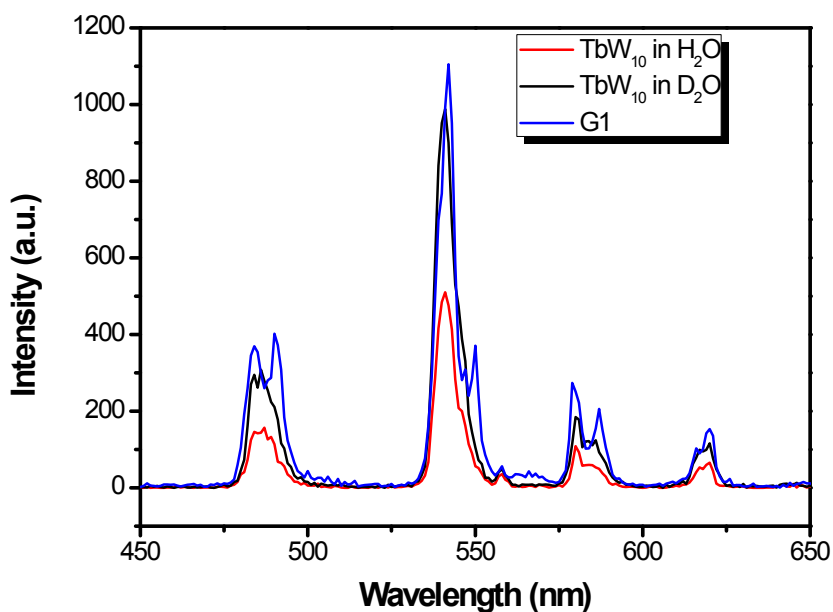


Fig. S6, Emission spectra of TbW₁₀ in H₂O (-), D₂O (-), and G1 primary solution (-) at an excitation wavelength of 280 nm.

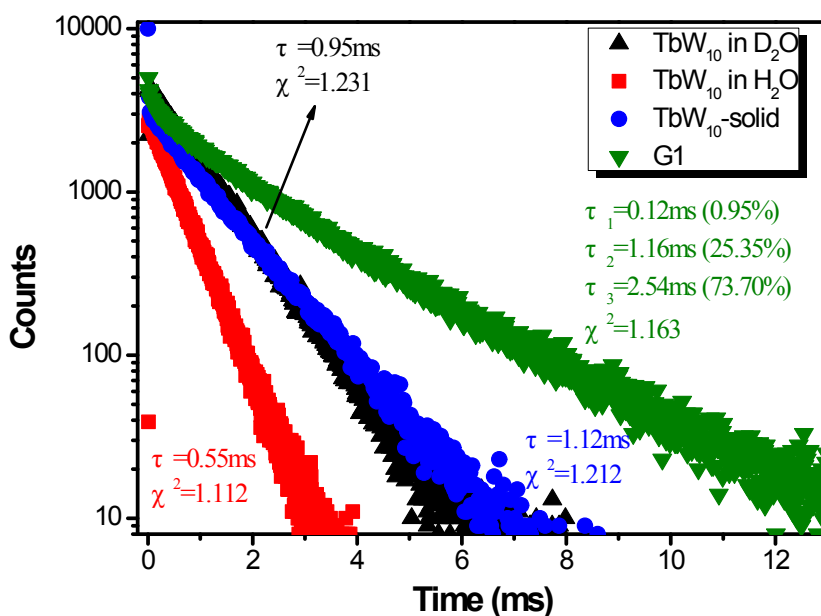


Fig. S7, Emission decay curves of TbW₁₀ in H₂O (■), D₂O (▲) and G1 (▼), and TbW₁₀ crystal (●) at an excitation wavelength of 280 nm. TbW₁₀ in H₂O and D₂O, and TbW₁₀ crystal was monitored at 542 nm, G1 was monitored at 587 nm.

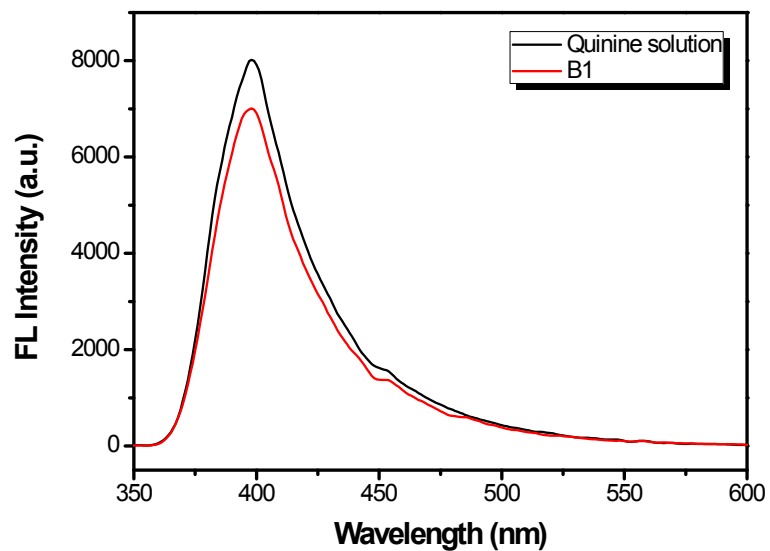


Fig. S8, Emission spectra of Quinine in H₂O (-) and B1 primary solution (-) at an excitation wavelength of 280 nm.

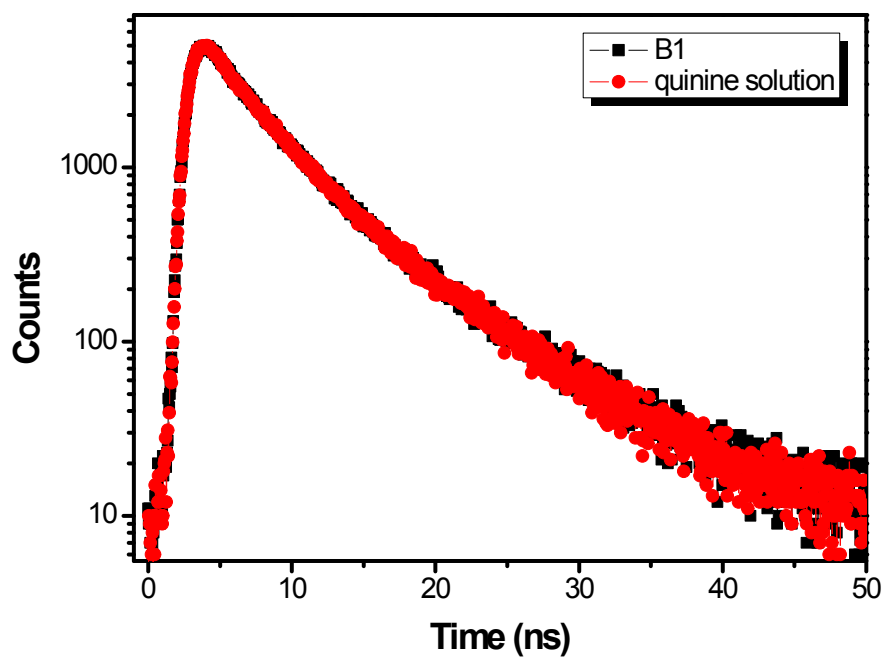


Fig. S9, Emission decay curves of B1 primary solution (■) and quinine solution (●) at an excitation

wavelength of 333 nm and monitored at 400 nm.

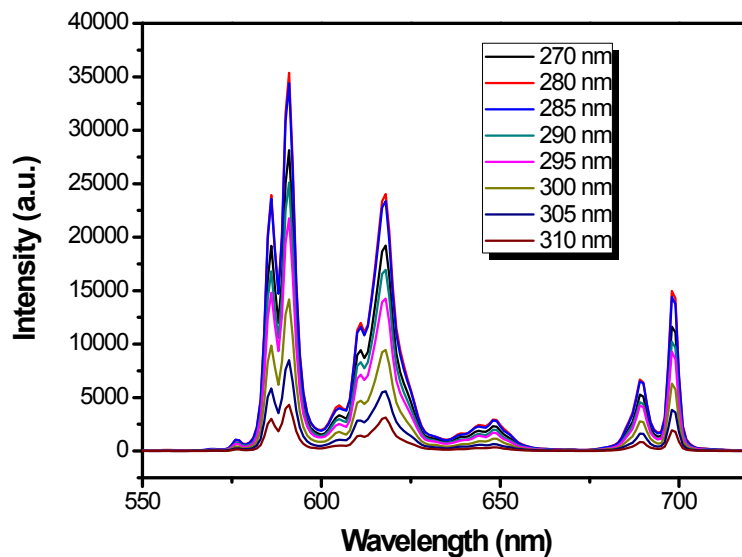


Fig. S10, Emission spectra of EuW₁₀ (3.50×10^{-5} M) complexed with PEO₁₁₄-*b*-PGu₂₃ (1.37×10^{-5} M) upon different excitation wavelengths.

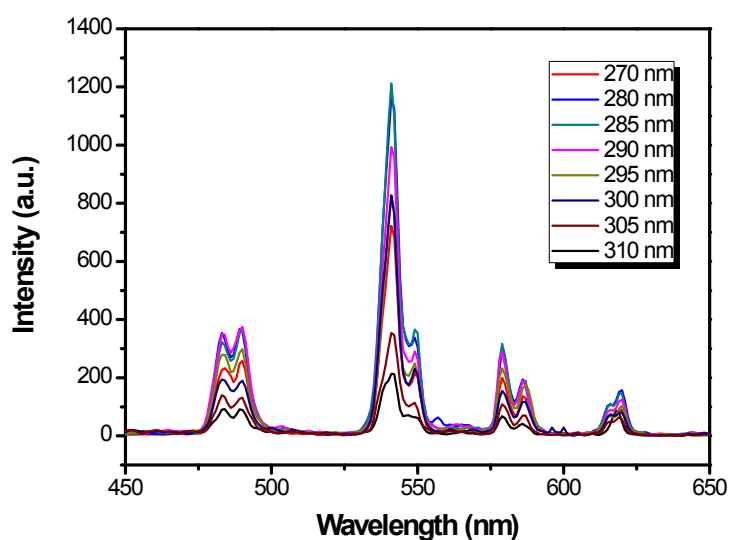


Fig. S11, Emission spectra of TbW₁₀ (3.50×10^{-5} M) complexed with PEO₁₁₄-*b*-PGu₂₃ (1.37×10^{-5} M) upon different excitation wavelengths.

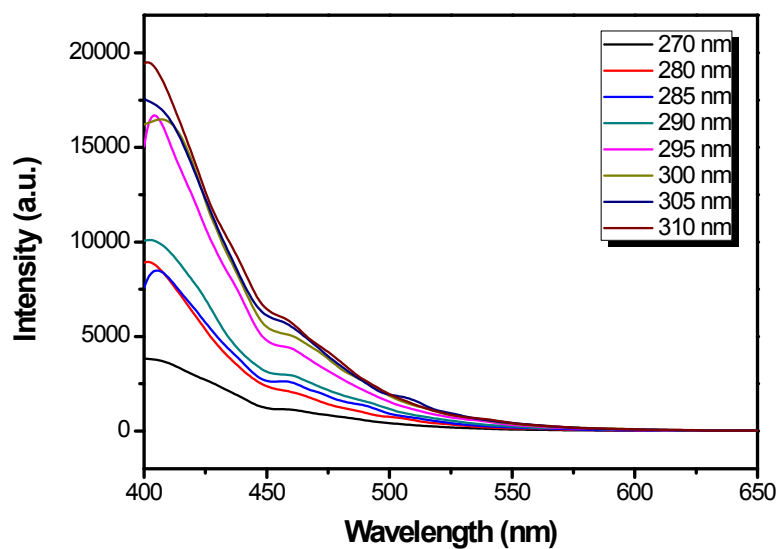


Fig. S12, Emission spectra of Quinine (3.50×10^{-5} M) complexed with PEO₁₁₄-*b*-PGu₂₃ (1.37×10^{-5} M) upon different excitation wavelengths.

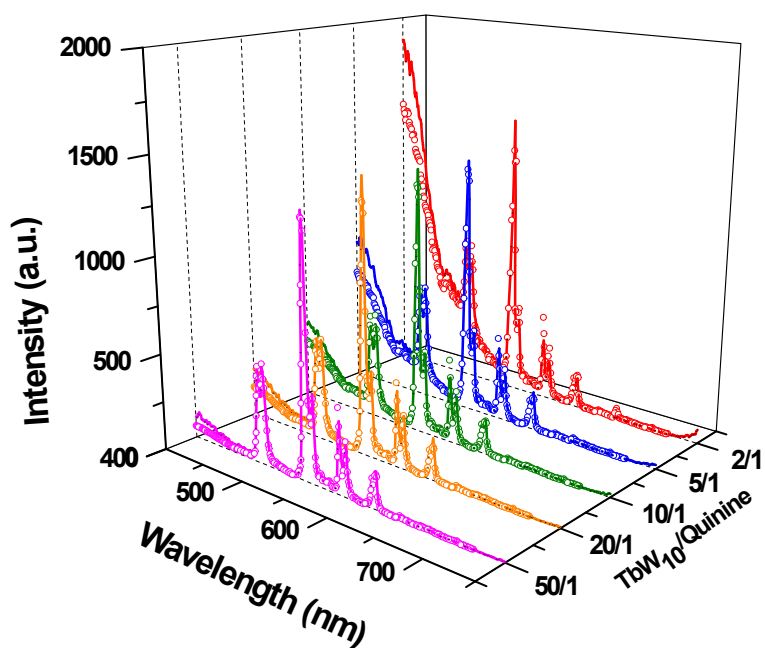


Fig. S13, Emission spectra of the hybrid solution mixtures TbW₁₀ - quinine series at different molar ratios at the excitation wavelength of 280 nm. Solid lines indicate the measured spectra and unfilled circles represent expected ones.

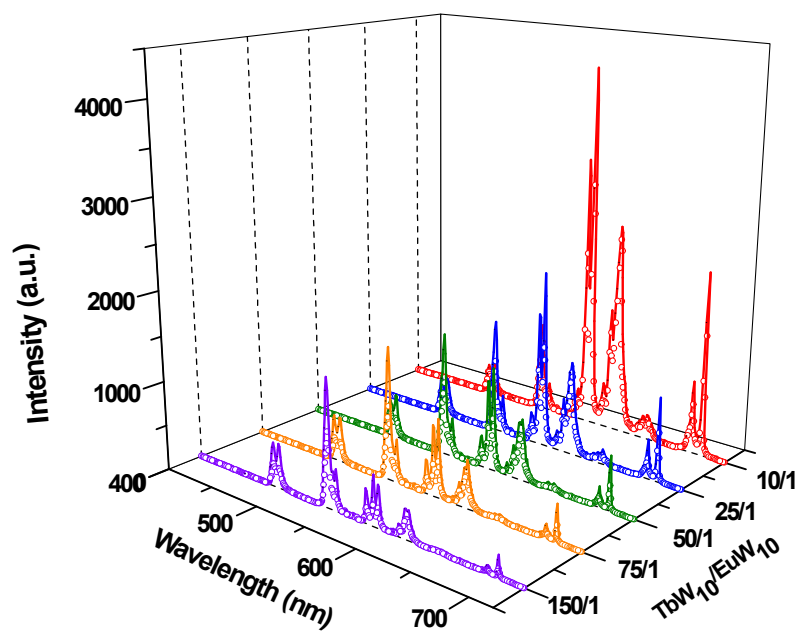


Fig. S14, Emission spectra of the hybrid solution mixtures EuW_{10} – TbW_{10} series at different molar ratios at the excitation wavelength of 280 nm. Solid lines indicate the measured spectra and unfilled circles represent expected ones.

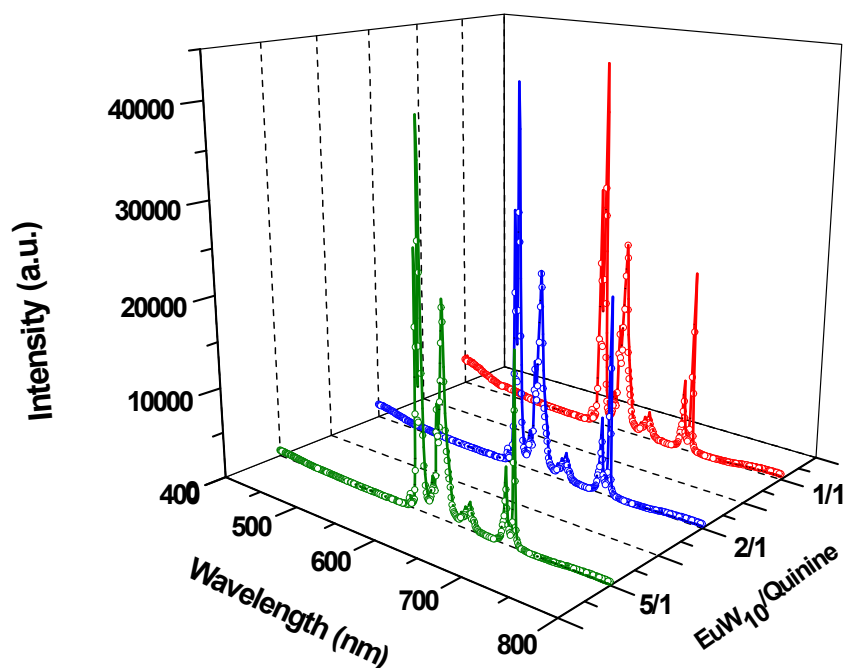


Fig. S15, Emission spectra of the hybrid solution mixtures EuW_{10} – Quinine series at different molar ratios at the excitation wavelength of 280 nm. Solid lines indicate the measured spectra and unfilled circles represent expected ones.

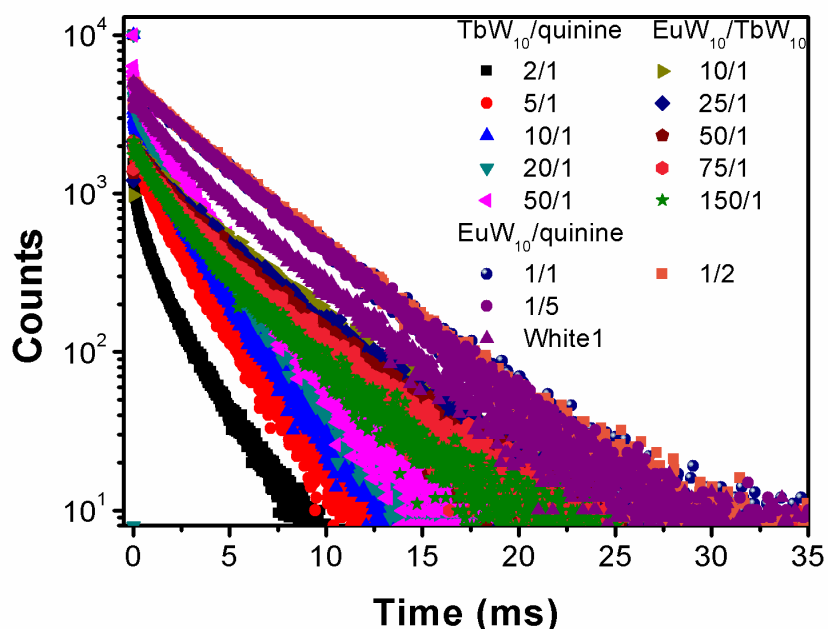


Fig. S16, Emission decay curves of TbW_{10} - quinine, EuW_{10} – quinine, and TbW_{10} – EuW_{10} series solutions at different ratios, and White1 solution excited at 280 nm and monitored at the co-emission wavelength 587 nm.

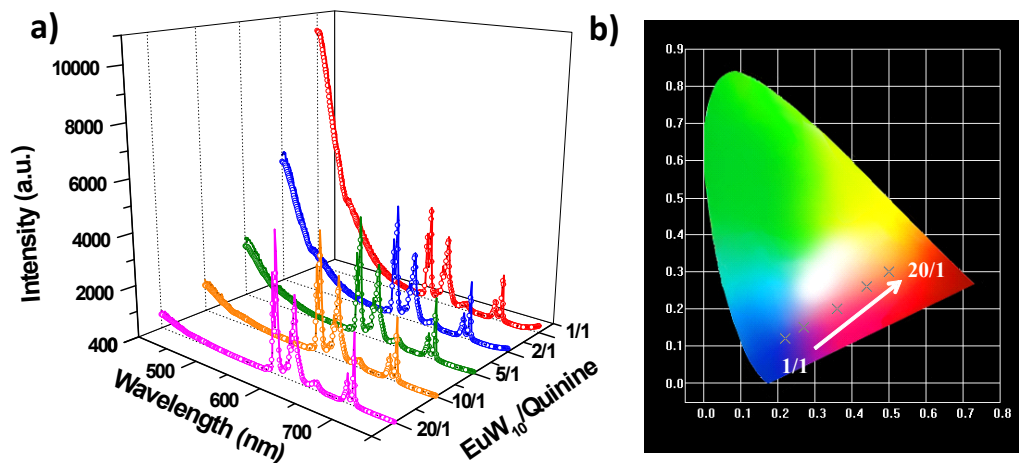


Fig. S17, a) Measured (solid line) and estimated (unfilled circles) emission spectra and b) Emission color coordinates of the EuW₁₀ – quinine series mixtures solutions at different ratios excited at 310nm.

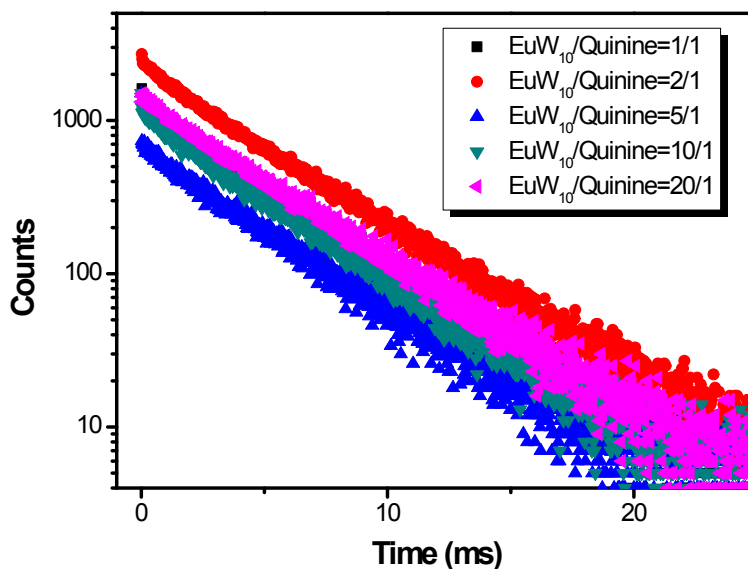


Fig. S18, Emission decay curves of EuW₁₀ – Quinine series solutions at different ratios excited at 310 nm and monitored at 587 nm.

Table S1, Fluorescent lifetimes and CIE chromaticity coordinates of EuW₁₀ – quinine series mixtures solutions at different ratios excited at 310 nm.

	EuW ₁₀ /Quinine				
	1/1	2/1	5/1	10/1	20/1
τ₁ (ms)	0.843	0.959	0.791	1.073	0.863
α₁ (%)	3.30	3.44	2.75	3.69	2.38
τ₂ (ms)	4.314	4.364	4.215	4.239	4.324
α₂ (%)	96.70	96.56	97.25	96.31	97.62
CIE (estimation)	(0.22, 0.12)	(0.27, 0.15)	(0.36, 0.21)	(0.44, 0.26)	(0.50, 0.31)
CIE (experiment)	(0.22, 0.12)	(0.27, 0.15)	(0.36, 0.20)	(0.44, 0.26)	(0.50, 0.31)

Table S2, Fluorescent lifetimes of TbW₁₀ – quinine, EuW₁₀ – quinine, and TbW₁₀ – EuW₁₀ – quinine series mixtures solutions at different excitation wavelengths.

λ _{ex} (nm)	TbW ₁₀ /Quinine				EuW ₁₀ /Quinine					TbW ₁₀ / EuW ₁₀ /Quinine						
	280	290	300	310	280	290	300	305	310	270	280	285	290	295	300	305
τ₁ (ms)	1.171	1.134	1.136	0.671	0.948	0.694	1.008	0.614	1.540	1.254	1.602	1.343	1.330	1.408	1.313	0.985
α₁ (%)	25.52	23.81	25.19	5.76	1.76	1.31	2.26	1.36	3.89	10.35	16.83	15.71	12.71	16.03	14.46	8.35
τ₂ (ms)	2.570	2.531	2.521	2.058	4.344	4.318	4.380	4.313	4.450	4.155	4.385	4.367	4.173	4.311	4.195	3.715
α₂ (%)	74.48	76.19	74.81	94.24	98.24	98.69	97.74	98.64	96.11	89.65	83.17	84.29	87.29	83.97	85.54	91.65

Table S3, ICP data of TbW₁₀ – EuW₁₀ series solution and White1. The concentration of the every solution was adjusted for precise measurement.

Sample	Tb (μg/ml)	Eu (μg/ml)	
TbW ₁₀ / EuW ₁₀	10/1	56.3492	5.3669
	25/1	67.3183	2.5538
	50/1	48.6238	0.9091
	75/1	59.0093	0.7512
	150/1	136.2037	0.8746
White1	52.1606	0.9281	

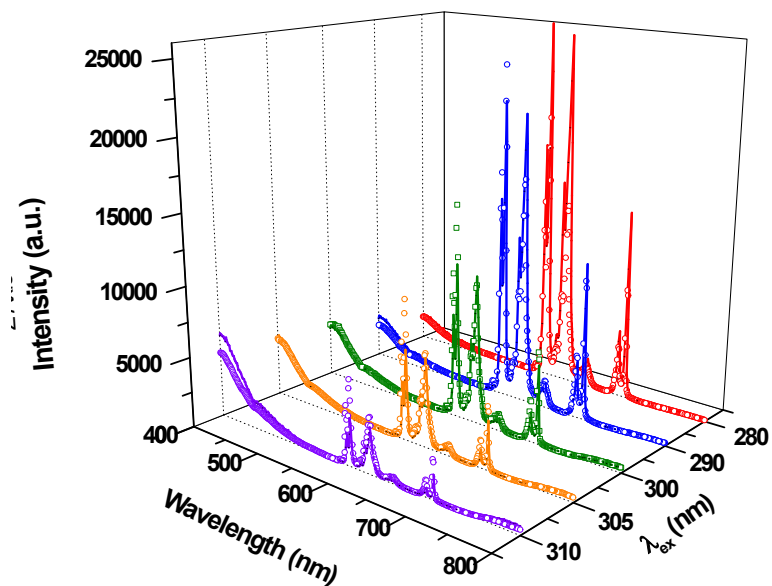


Fig. S19, Emission spectra of $\text{EuW}_{10}/\text{quinine} = 3/1$ hybrid solution mixture upon different excitation wavelengths. Solid lines indicate the measured spectra and unfilled circles represent expected ones.

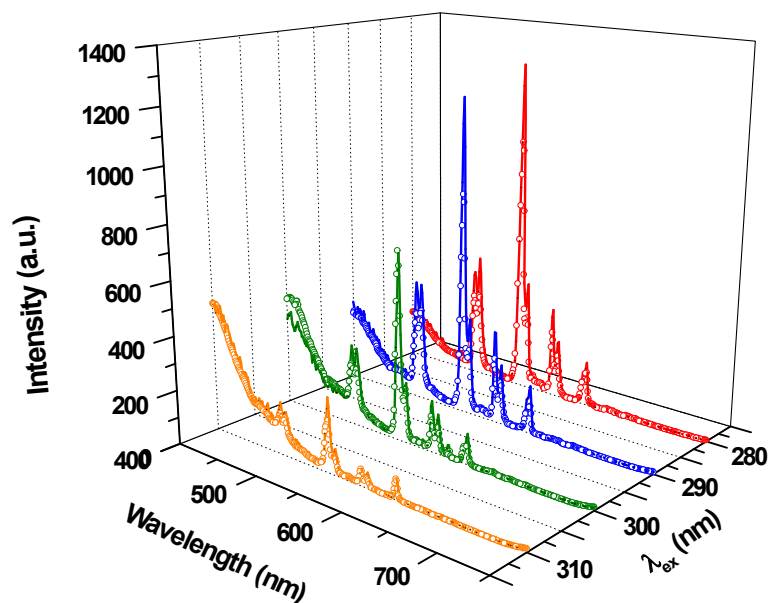


Fig. S20, Emission spectra of $\text{TbW}_{10}/\text{quinine} = 30/1$ hybrid solution mixture upon different

excitation wavelengths. Solid lines indicate the measured spectra and unfilled circles represent expected ones.

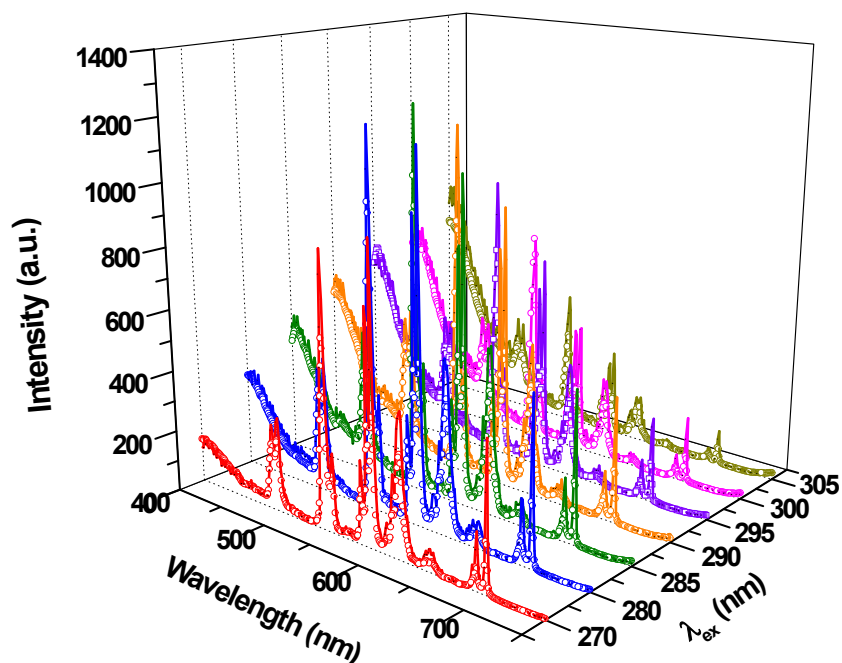


Fig. S21, Emission spectra of TbW₁₀/EuW₁₀/quinine = 50/1/3 hybrid solution mixture upon different excitation wavelengths. Solid lines indicate the measured spectra and unfilled circles represent expected ones.

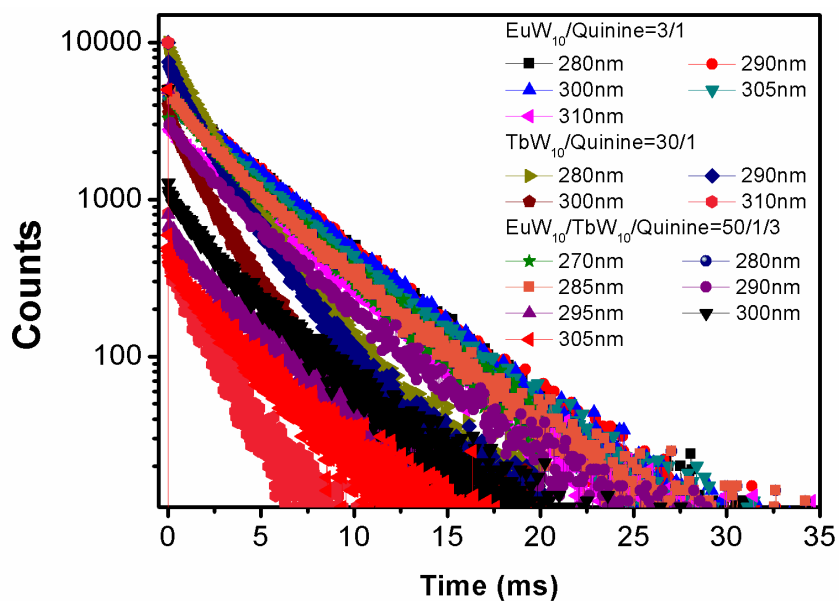


Fig. S22, Emission decay curves of $\text{EuW}_{10}/\text{Quinine} = 3/1$, $\text{TbW}_{10}/\text{Quinine} = 30/1$, and $\text{EuW}_{10}/\text{TbW}_{10}/\text{Quinine} = 50/1/3$ solutions excited at different Wavelengths and monitored at 587 nm.

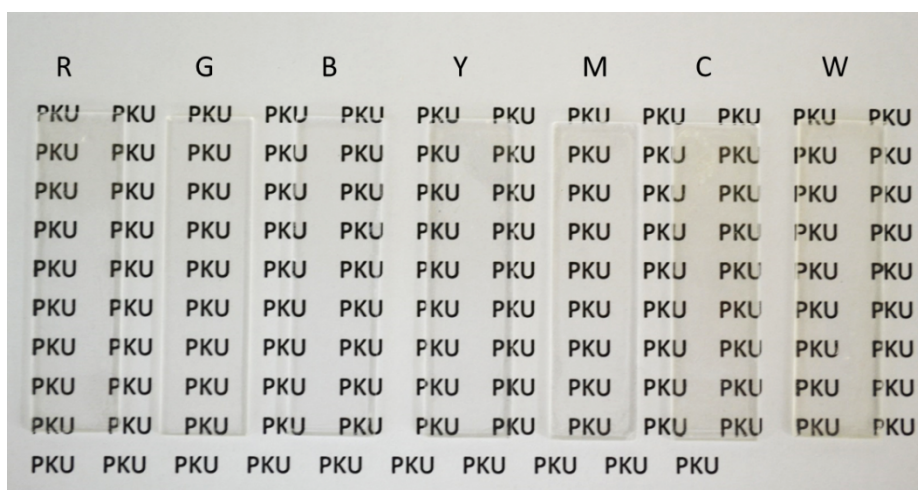


Fig. S23, Photographs of representative films under daylight.

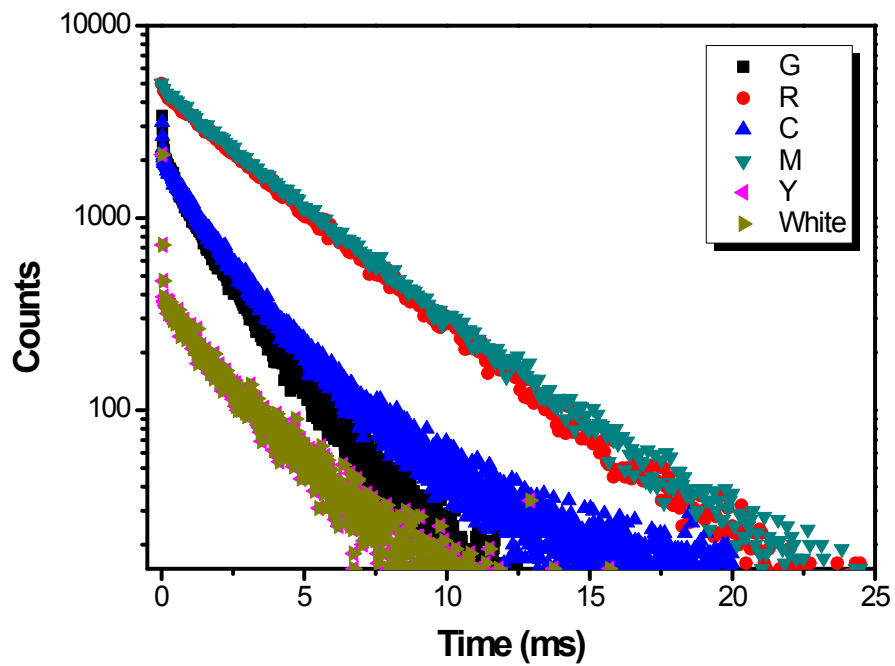


Fig. S24, Emission decay curves of representative films excited at 280 nm.