Supplementary Information

Design and Synthesis of RNA-Responsive o-Phenanthroline Eu(III) complexes Probes for STED Super-Resolution Dual-Targeted Bioimaging

Hao Yuan,[a] Tao Wang,[b] Tong Zhu,[b] Zhihui Feng,[b] Fei Wang,[b] Yupeng Tian,[b] Xiaohe Tian [a c d]

a Department of Radiology and National Clinical Research Center for Geriatrics, Huaxi MR Research Center (HMRRC), Functional and Molecular lmaging Key Laboratory of Sichuan Province, West China Hospital, Sichuan University, Chengdu, China.

b College of Chemistry and Chemical Engineering, Key Laboratory of Functional Inorganic Material Chemistry of Anhui Province, Anhui University, Hefei 230601, PR China.

c. Orthopedics Department of Zunyi Medical University Affiliated Hospital

d. School of Basic Medicine, Kunming Medical University

Experimental Section

1. Materials and Apparatus

All chemical reagents and organic solvents were purchased from Aladdin Industries and Mcleaned Reagent Network, and were dried and purified according to standard methods. The 1H-NMR and 13C-NMR spectra were recorded on Bruker AV 400 spectrometer (Germany). Mass Spectrometer was recorded using Bruker Autoflex III TOF/TOF. UV-vis absorption spectra were performed by the UV-265 spectrophotometer. Fluorescence was measured on a Hitachi F-7000 spectrofluorometer. Confocal and STED imaging data acquisition and processing were performed using Leica SP8 microscopy, 100/63×oil-immersion objective lens and and Image J.

1.1 Synthesis and characterization

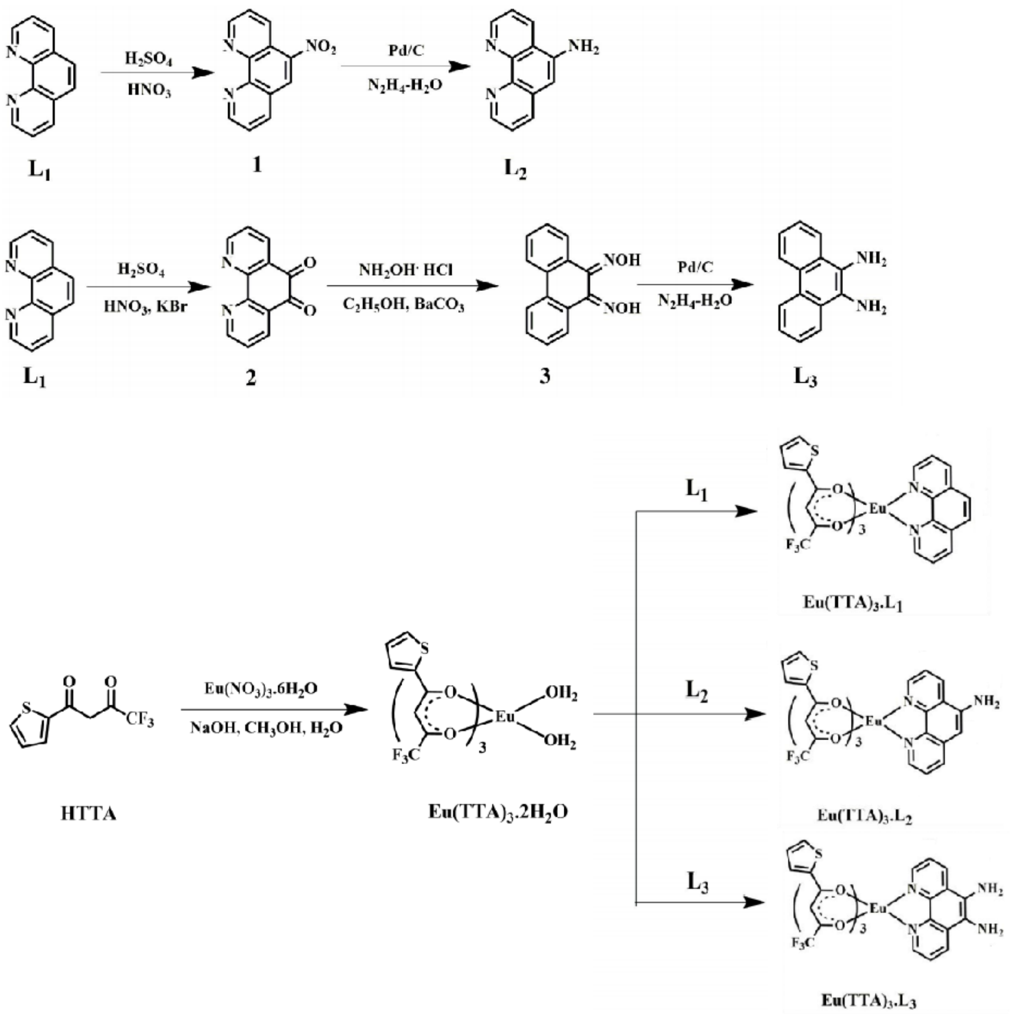

Figure S1 Synthetic routes of Eu(TTA)3-L1-3.

➢ Synthesis of $Eu(TTA)_3$-H_2O

Intermediates L2, L3 were synthesized according to Ref. [1, 2].Weigh 6.660 g (30 mmol) of HTTA in a 100 mL flask, dissolve it with an appropriate amount of methanol, add to it 1.200 g (30 mmol) of NaOH solution dissolved in distilled water, stirred for 5 min, and added 2 ml of methanol to the flask. 4.460 g (10 mmol) of $Eu(NO_3)_3$-$6H_2O$ solution dissolved in 2 ml of methanol was added to the flask, and the reaction was stirred at room temperature for 24 h. 200 mL of distilled water was added to the reaction solution with constant stirring. The reaction solution was stirred for 24 h at room temperature, and 200 mL of distilled water was added to the reaction solution with constant stirring, and a large amount of white solid was generated, filtered, washed and dried under vacuum to obtain a white powder The solid was 8.64 g, yield: 91%. ^{1}H-NMR (d6-DMSO, 400 MHz, ppm)δ: 7.74-7.20 (m, 4H), 6.52 (s, 4H), 6.35 (s, 3H), 4.63-4.21 (m, 1H), 3.78 (s, 3H), 1.91 (s, 4H); ^{13}C-NMR (d6-DMSO, 100MHz, ppm)δ: 181.53, 135.46, 130.21, 128.92, 124.65, 97.21, 38.91.

➢ Synthesis of Eu(TTA)$_3$-L1-3

Weigh 0.2556 g (0.5 mmol) of Eu(TTA)$_3$-2H_2O in a 100 mL flask, dissolve the appropriate amount of $CHCl_3$ The reaction solution was added with 0.5 mol of ligand L_{1-3} solution dissolved with appropriate amount of $CHCl_3$, heated to reflux. The reaction solution was heated to reflux and reacted for 12 h. After cooling and standing, a light yellow solid was precipitated, filtered, washed and dried. The complex Eu(TTA)$_3$-L1-3 was obtained.

Eu(TTA)$_3$-L1 yield: 85%. ^{1}H-NMR(d6-DMSO, 400 MHz, ppm)δ: 9.09 (s, 2H), 8.50 (s, 2H), 7.89 (d, J = 88.7 Hz, 3H), 7.42 (s, 4H), 6.57 – 6.24 (m, 6H), 3.75 (s, 3H). ^{13}C-NMR (100MHz, DMSO-d_6), δ(ppm): 170.23, 159.91, 156.71, 143.29, 143.24, 127.75, 127.71, 114.06 ◦ ESI-MS: m/z,$[C_{36}H_{20}EuF_9N_2O_6S_3]^+$=cal: 995.507, found: 995.72.

Eu(TTA)$_3$-L2 yield: 84%. ^{1}H-NMR(d6-DMSO, 400 MHz, ppm)δ: 8.62 (d, J = 8.5 Hz, 1H), 8.43 (d, J = 4.4 Hz, 2H), 8.13 – 7.97 (m, 5H), 7.72 (dd, J = 5.0, 1.1 Hz, 3H), 7.67 – 7.60 (m, 1H), 7.50 (dd, J = 8.4, 4.3 Hz, 1H), 7.42 (s, 3H), 7.16 (dd, J = 5.0, 3.8 Hz, 3H), 6.84 (s, 1H), 5.74 (d, J = 8.3 Hz, 1H). ^{13}C-NMR (100MHz, DMSO-d_6), δ(ppm): 171.32, 162.77, 160.24, 134.53, 134.50, 131.42, 131.33, 129.01, 128.98, 128.58, 128.52, 124.57, 124.53, 123.67, 123.56, 116.40, 116.18。ESI-MS: m/z,$[C_{36}H_{21}EuF_9N_3O_6S_3]^+$=cal: 1010.513, found: 1010.745.

Eu(TTA)$_3$-L$_3$ yield: 86%. ^{1}H-NMR(d6-DMSO, 400 MHz, ppm)δ: 9.05 (d, J = 4.0 Hz, 2H), 8.05 (d, J = 8.2 Hz, 2H), 7.49 (dd, J = 25.2, 4.6 Hz, 6H), 6.50 (s, 5H), 6.14 (s, 3H), 3.73 (s, 4H). ^{13}C-NMR (100MHz, DMSO-d_6), δ(ppm): 171.35, 141.86, 135.59, 129.99, 129.01, 128.28, 126.33。ESI-MS: m/z,$[C_{36}H_{22}EuF_9N_4O_6S_3]^+$=cal: 1025.519, found: 1025.753.

1.2 Crystal Structure

Crystals of Eu(TTA)$_3$-L2 were obtained using the solvent volatilization method: Equal amounts of Eu(TTA)$_3$-L2 solid powder were dissolved in 5 mL of acetonitrile and ethanol solvent, respectively, and the two were mixed. Filtered and evaporated at room temperature for 72 h to obtain rod-shaped yellow transparent crystals. The crystallographic data were analyzed using X-ray single crystal diffractometer and the crystallographic data obtained were resolved by OLEX2 software. As shown in the table below, the Eu-N bond length in the crystal structure is about 2.57 Å and the Eu-O bond length is about 2.37 Å.

1.3 Two-photon excited fluorescence (TPEF) spectroscopy

Using a fully automated femtosecond titanium gemstone pulsed laser: sapphire system (680-1080 nm, 80 MHz, 140 fs), The two-photon excitation fluorescence (2PEF) method was used to obtain two-photon absorption cross sections (2PA) for Eu(TTA)$_3$-

$2H_2O$ and Eu(TTA)$_3$-L1-3. Two-photonaction cross section σ values were determined by the following equation:

$$\sigma = \sigma_{ref} \frac{\Phi_{ref}}{\Phi} \frac{c_{ref}}{c} \frac{n_{ref}}{n} \frac{F}{F_{ref}}$$

The subscript "ref" represents the reference data of rhodamine in DMSO solution (c=1.0×10-3mol/L, with a 1 cm standard quartz cell). "F" is the intensity of the fluorescence signal detected by the spectrometer. "n", "c" and "Φ" are the refractive index of the solution, the concentration of the solution and the quantum yield of fluorescence, respectively.

1.4 Three-photon excitation fluorescence (3PEF) measurement method

A control was performed using Rhodamin 6G as a standard sample. The relative fluorescence intensity of the specimens was calculated. All test cuvettes were: 1cm optical length quartz liquid cell. Spectrometer: Ocean Optics QE65 Pro (300-2500 nm), laser source: Coherent Astrella+TOPAS Prime (1100-1800) nm, 1 kHz, 120 fs. The three-photon absorption cross section was calculated from the following equation:

$$\sigma_S^{(3)} = \frac{c_R \cdot n_S \cdot F_S(\lambda) \cdot Q_R}{c_S \cdot n_R \cdot F_R(\lambda) \cdot Q_S} \cdot \sigma_R^{(3)}$$

where the subscript "R" represents the reference data of rhodamine in DMSO solution (c=1.0×10-3mol/L), and the subscript S represents the parameters of the assay sample. "C", "n", "F(λ)", and "Q" are the concentration, refractive index, intensity of fluorescence signal, and fluorescence quantum yield, respectively.

1.5 Circular dichroism analysis

Add 0-10 μM $Eu(TTA)_3$-L3 ($c=1.0\times10^{-5}$ mol/L, in ultrapure water) solution to 1 mL RNA (c=10 μM/L, in ultrapure water) solution dropwise, mix thoroughly and measure the absorption coefficient of the solution by circular dichroism (BRIGHTTIME Chirascan, JASCO810, Jasco-815).

1.6 Cell culture

The Hela cells were cultured in 25 cm^2 culture flasks in DMEM, supplemented with fetal bovine serum (10 %), penicillin (100 units/mL) and streptomycin (50 units/mL) at 37 °C in a CO_2 incubator (95 % relative humidity, 5 % CO_2). Cells were seeded in 35 mm glass bottom cell culture dishes, at a density of 1×10^5 cells.

1.7 Cytotoxicity assay

The toxicity of different concentrations of $Eu(TTA)_3$-L1-3 on human cervical cancer (Hela) cells was determined by MTT method. (1) Hela cells were cultured in DMEM with 10% calf serum at 37°C and 5% CO2 for 48 hours. (2) Hela cells were inoculated in 96-well plates at an inoculum concentration of 1×10^4 cells/100 μL and incubated at 37°C, 5% CO_2 for 48 hours. (3) The medium was aspirated and 100 μL of calf serum-free DMEM medium was re-added to each well, and different concentrations of $Eu(TTA)_3$-L1-3 (DMSO) were added to the wells, and 7 parallel controls were set up for each concentration gradient. The incubation was continued for 8 hours under the same conditions as above. (4) Add 50 μl MTT (5 mg/mL) to each well and incubate for 4 hours under the same conditions as described above. (5) After aspirating the medium with a 1 mL syringe, 200 μl of LDMSO was added to each well. After shaking the plate using a Tecan Infinite M200 multifunctional enzyme marker, the absorbance at 570 nm was measured, and the cell viability of cells in the wells was calculated using the wells without the sample added as a control with 100% cell viability.

1.8 Photostability analysis

Under the imaging conditions of STED (λex=405nm, λem=610nm, doughnut laser: is 660nm) at a scanning rate of once per second for 500 seconds of cyclic irradiation.

1.9 STED super-resolution image.

The compound was excited under STED laser (doughnut laser: 660 nm), the emission signals were collected using HyD reflected light detectors (RLDs) with 2048*2048 pixel and *100 scanning speed. The STED micrographs were further processed 'deconvolution wizard' function using Huygens Professional software (version: 16.05) under authorized license. The area radiuses were estimated under 0.02 micros with the exclusion of 60 absolute background values. Maximum iterations were 10-time, signal-to- noise ration 30 was applied, with quality threshold 0.02; iteration mode: Optimized; Brick layout:Auto.

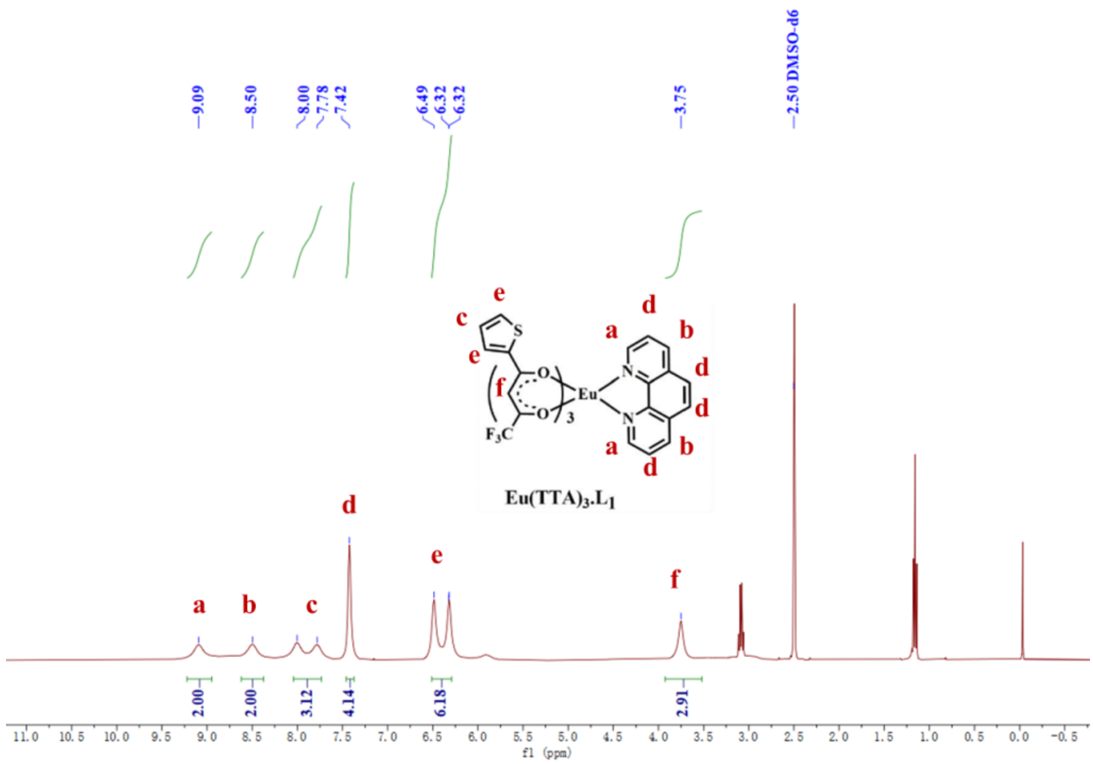

Figure S2 ^{1}H-NMR spectrum of $Eu(TTA)_3 \cdot L1$

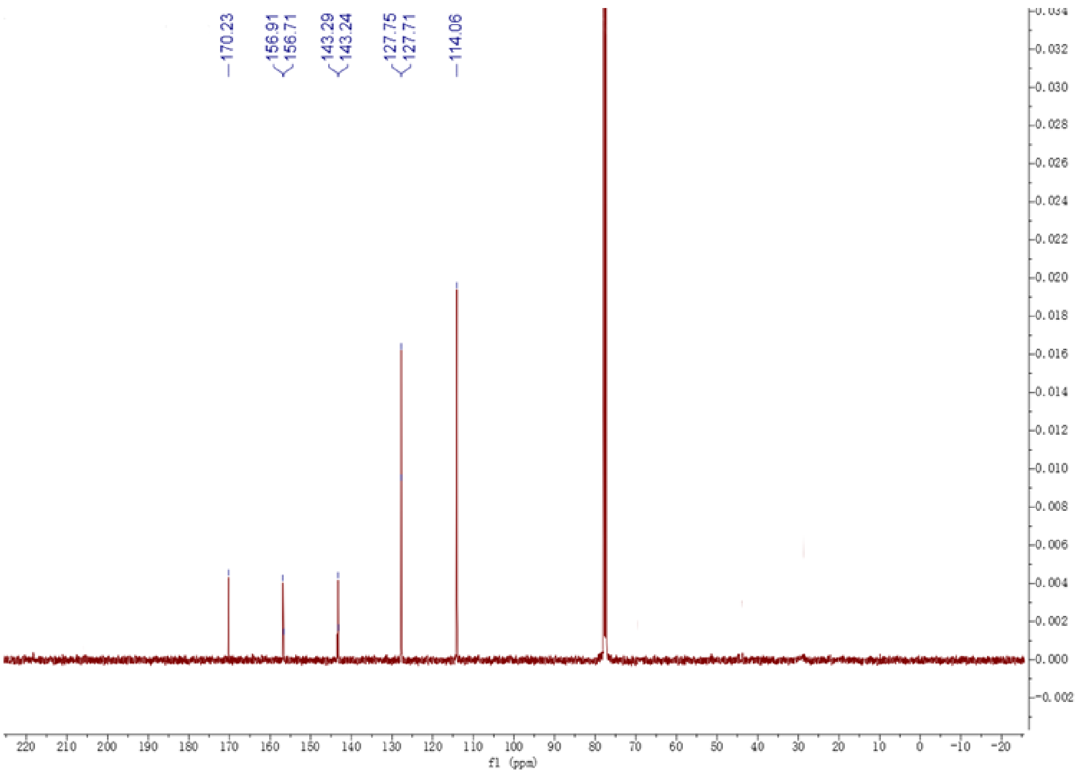

Figure S3 ^{13}C-NMR spectrum of $Eu(TTA)_3 \cdot L1$

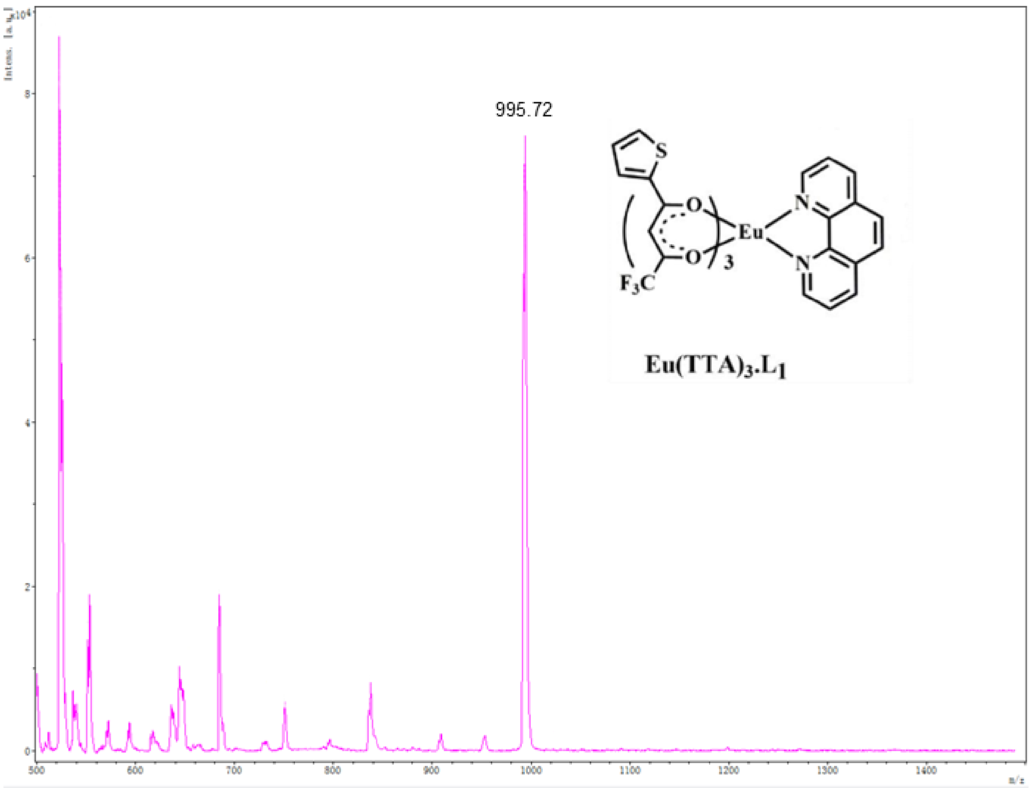

Figure S4 Maldi-tof-MS. of Eu(TTA)$_3$·L1

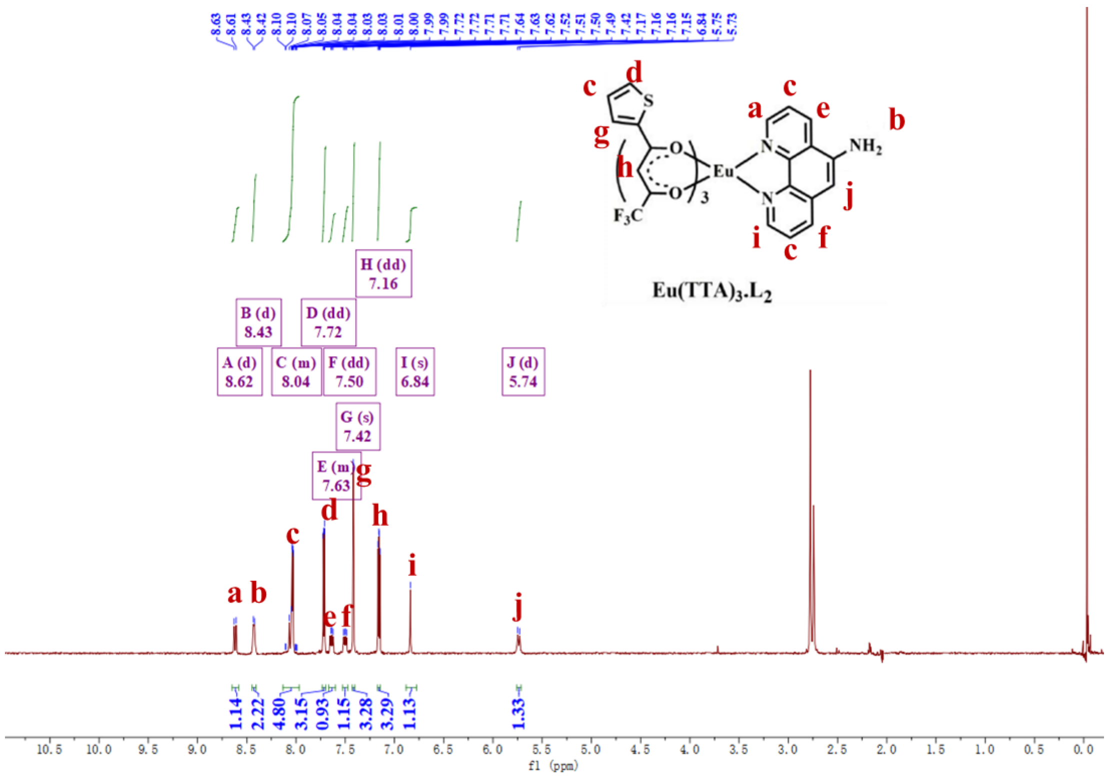

Figure S5 ^{1}H-NMR spectrum of Eu(TTA)$_3$·L2

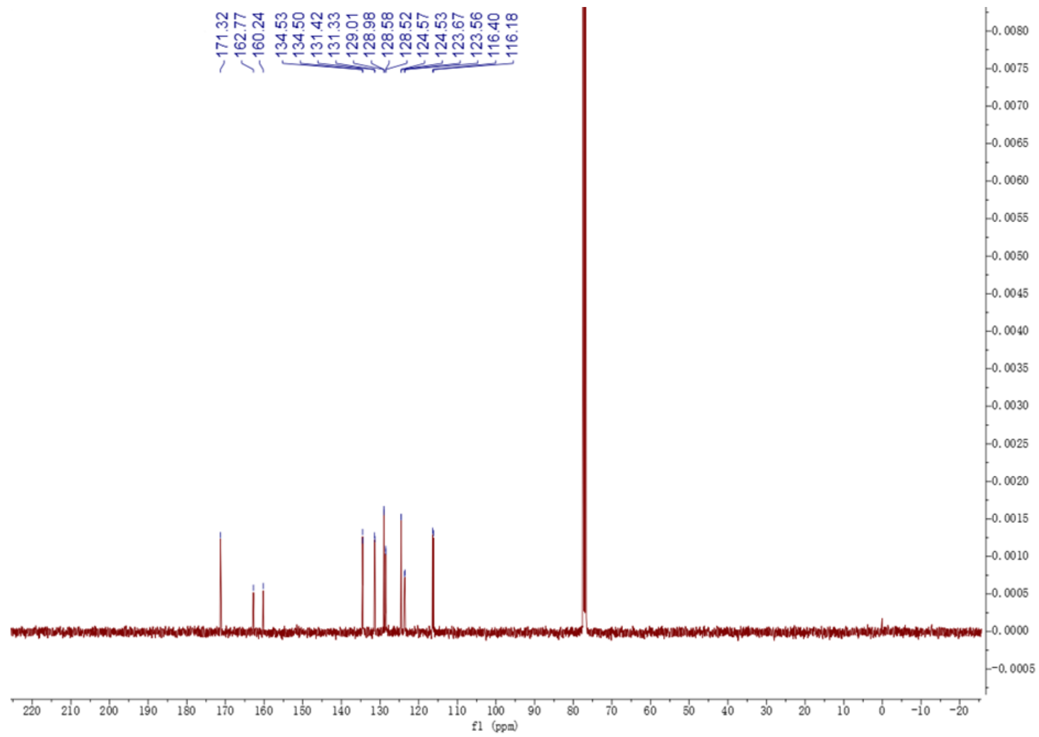

Figure S6 ^{13}C-NMR spectrum of Eu(TTA)$_3$·L2

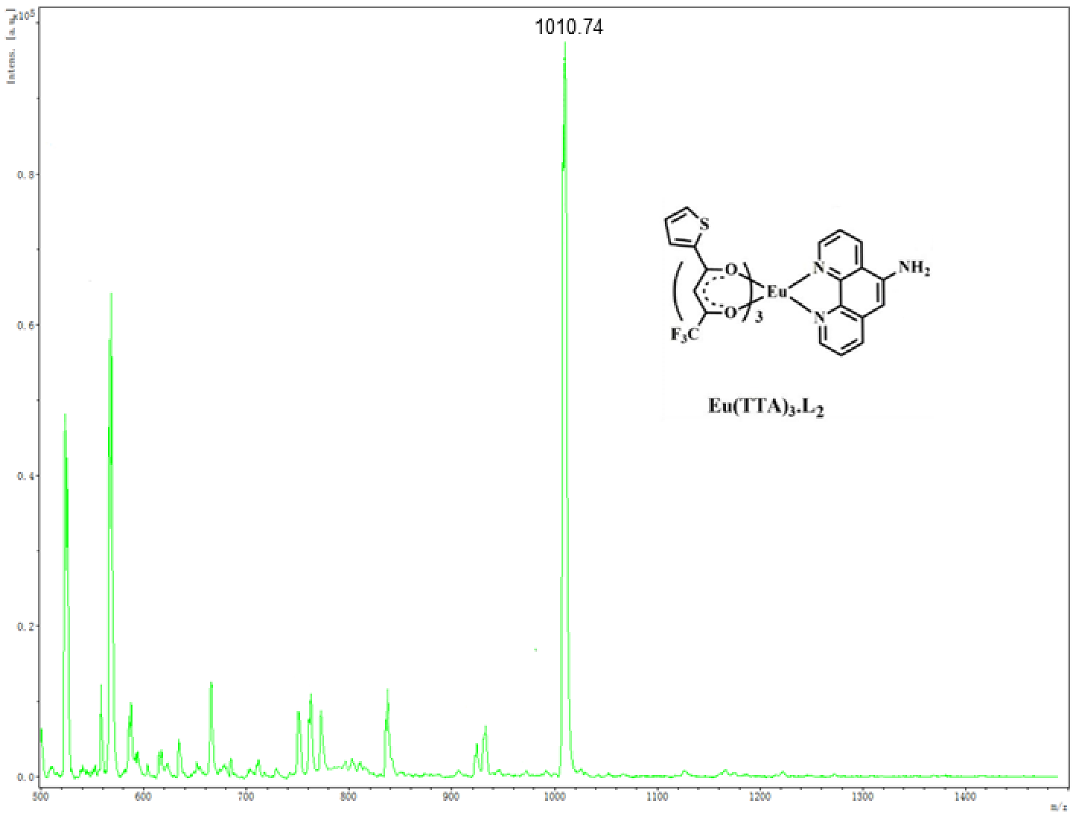

Figure S7 Maldi-tof-MS. of Eu(TTA)$_3$·L2

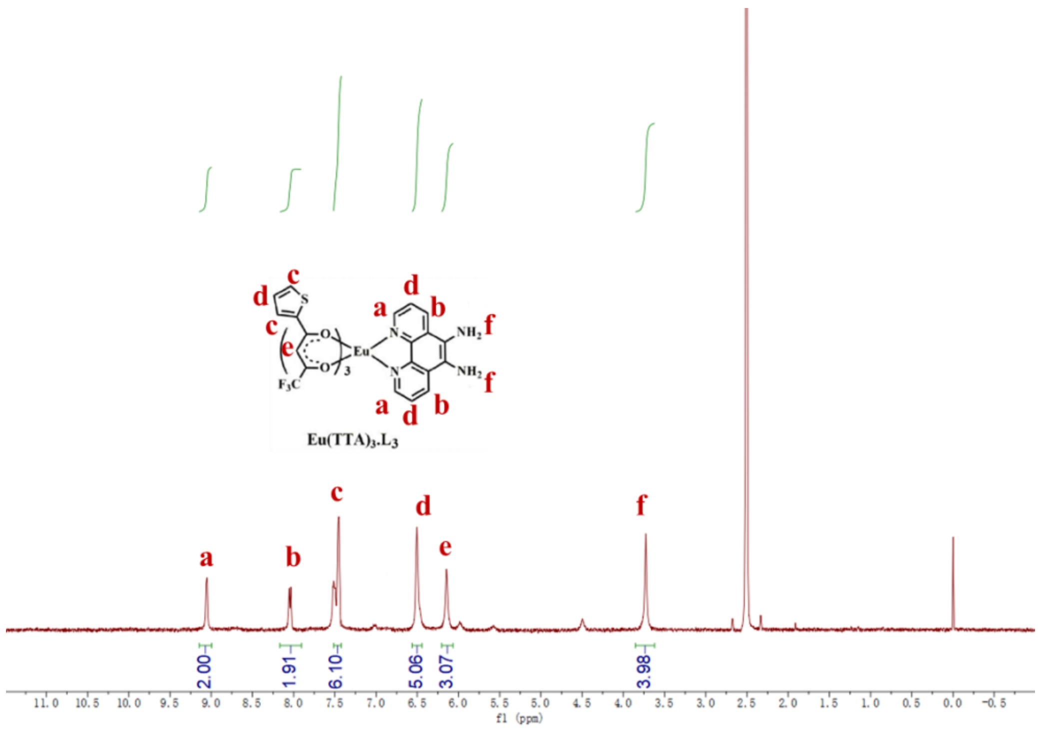

Figure S8 ^{1}H-NMR spectrum of $Eu(TTA)_3 \cdot L3$

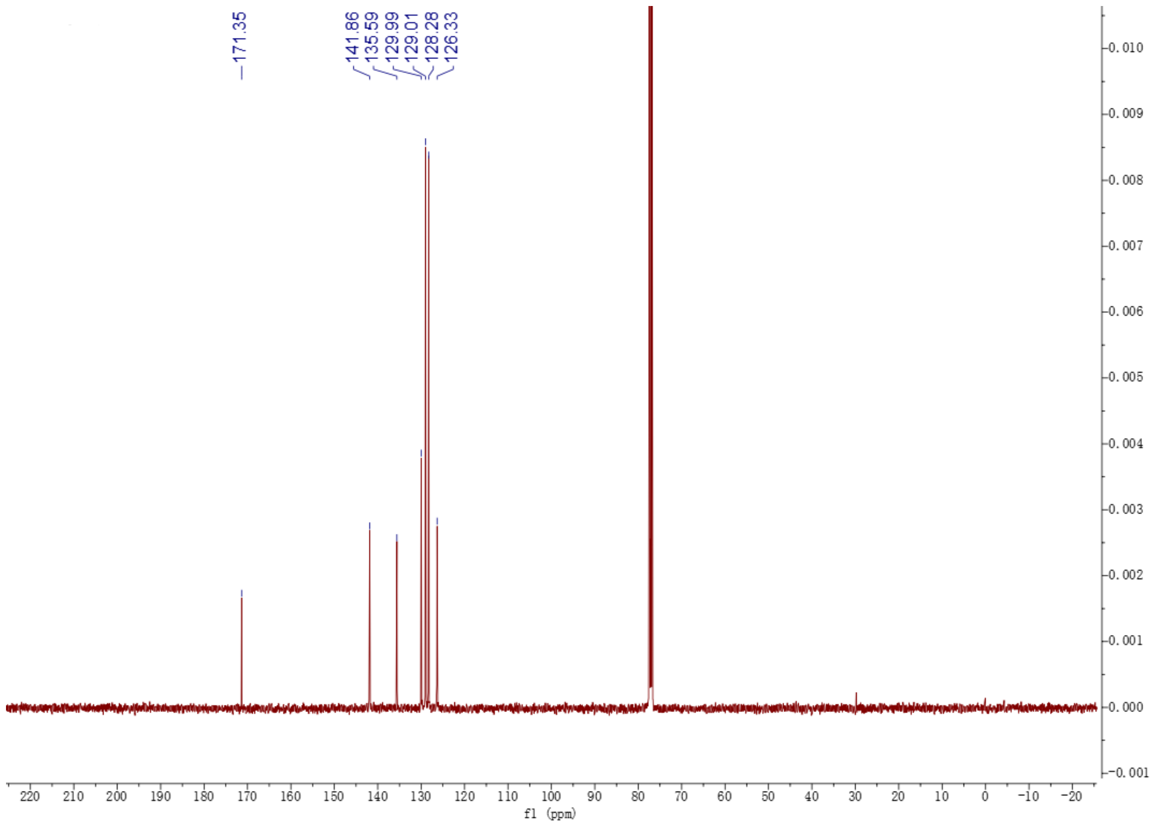

Figure S9 ^{1}C-NMR spectrum of $Eu(TTA)_3 \cdot L3$

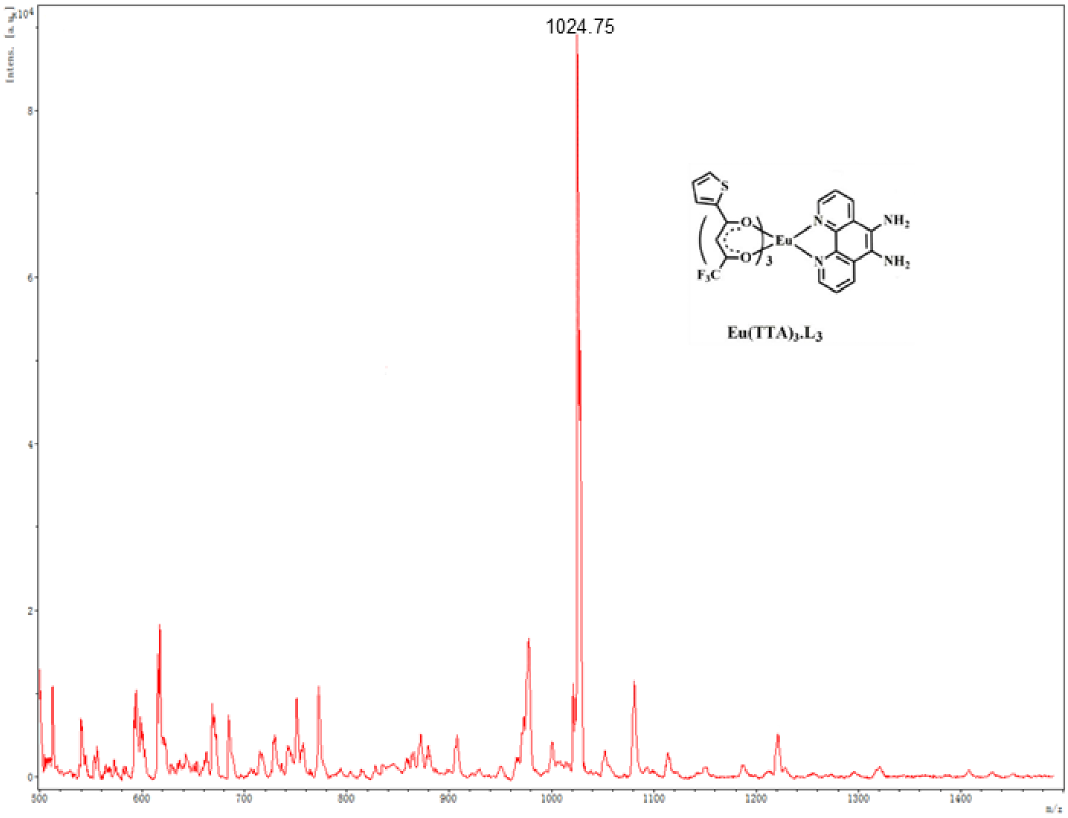

Figure S10 Maldi-tof-MS. of Eu(TTA)$_3$·L3

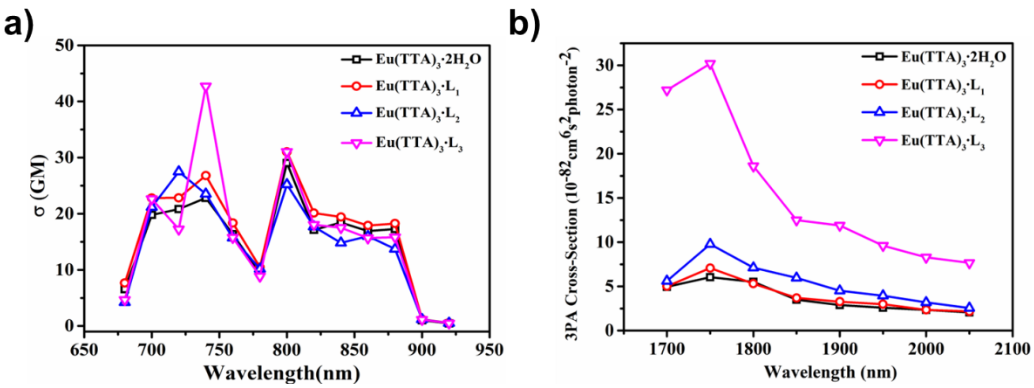

Figure S11 Two-photon absorption cross-sections (a) and three-photon absorption cross-sections (b) of Eu(TTA)$_3$·L1-3 in DMSO. (c=1.0×10-3 mol/L)

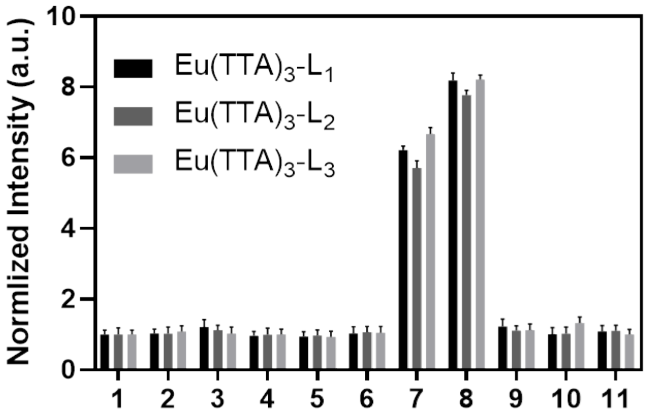

Figure S12 Fluorescence responses of Eu(TTA)$_3$·L1-3 (10 μM) to (1) blank, (2) Ca^{2+}, (3) Mg^{2+}, (4) ATP, (5) glutathione, (6)HClO, (7) RNA, (8) liposome, (9) H_2O_2, (10) DNA, (11) glycerol

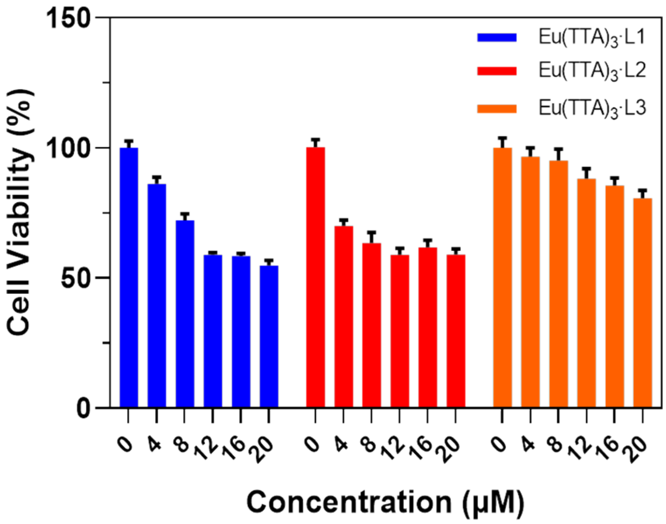

Figure S13 MTT assay: using Eu(TTA)$_3$·L1-3(0, 4, 8, 12, 16, and 20μM) against HeLa cells for incubation 8h

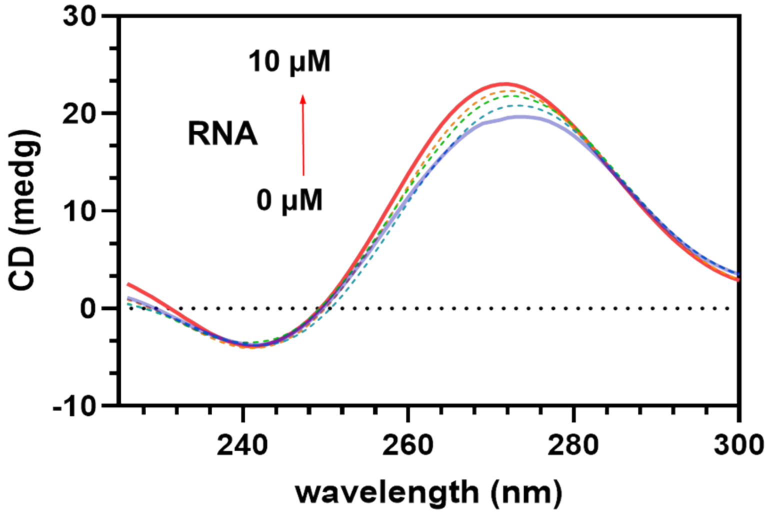

Figure S14 CD profiles of Eu(TTA)$_3$-L3 in RNA(c=1.0×10^{-5} mol/L) at concentrations of 0 ~ 10 μM.

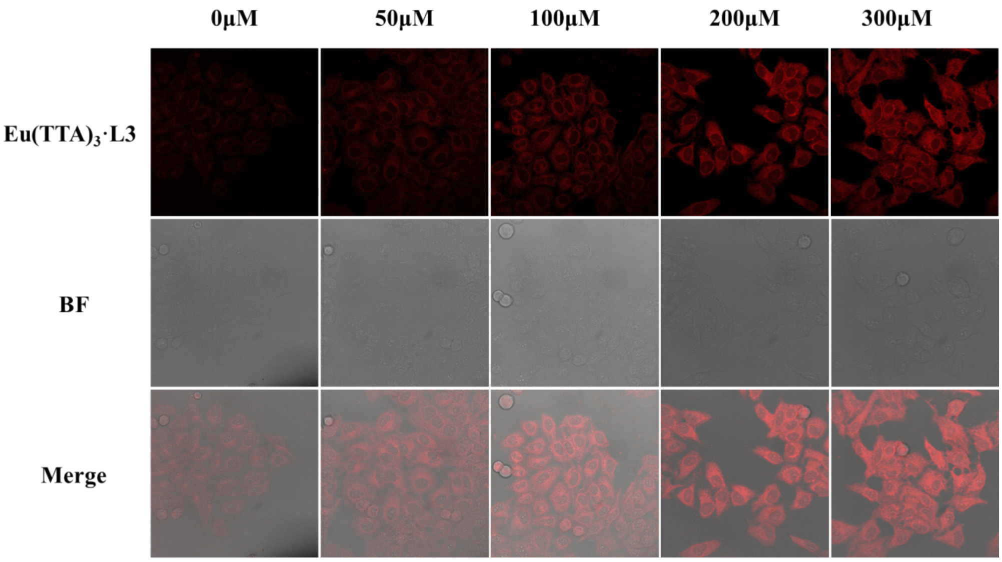

Figure S15 Response of Eu3(10 μM) to different concentrations of exogenous RNA in Hela-fixed cells.

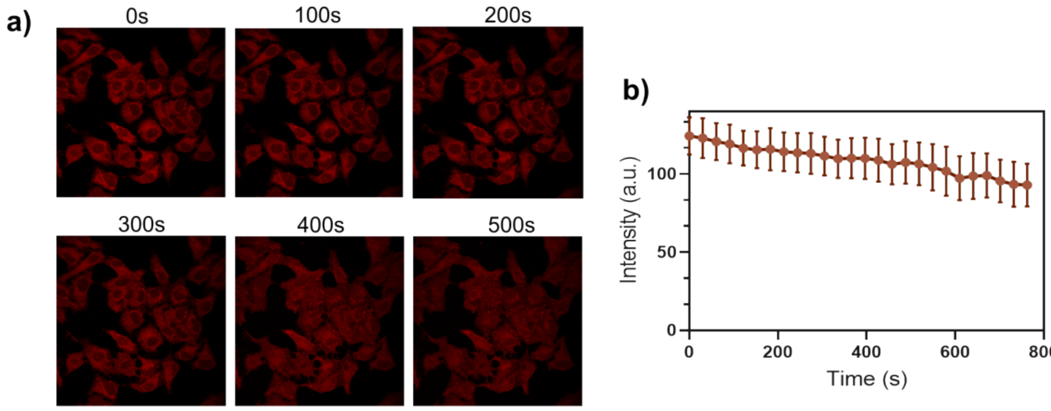

Figure S16 Photostability experiments. (a) Eu3(10 μM) were monitored for cellular photostability by STED. (b) Quantification of fluorescence intensity in (a).

Table S1 Crystal data of $Eu(TTA)_3{\cdot}L2$

Complexes	$Eu(TTA)_3{\cdot}L_2$
Identification code	a
Empirical formula	$C_{72}Eu_2F_{18}N_6O_{12}S_6$
Formula weight	1979.06
Temperature/K	296.15
Crystal system	triclinic
Space group	P1
a/Å	9.6768(14)
b/Å	13.379(2)
c/Å	15.458(2)
α/°	92.611(2)
β/°	93.941(2)
γ/°	103.219(2)
Volume/Å^3	1939.9(5)
Z	1
ρ_{calc}g/cm^3	1.694
μ/mm^{-1}	1.869
F(000)	954.0
Crystal size/mm^3	? × ? × ?
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	2.646 to 54.764
Index ranges	$-12 \le h \le 12$, $-17 \le k \le 16$, $-19 \le l \le 19$
Reflections collected	15205
Independent reflections	11876 [R_{int} = 0.0361, R_{sigma} = 0.1012]
Data/restraints/parameters	11876/3105/1045

Goodness-of-fit on F^2	1.031
Final R indexes [I>=2σ (I)]	R_1 = 0.0783, wR_2 = 0.1905
Final R indexes [all data]	R_1 = 0.1263, wR_2 = 0.2338
Largest diff. peak/hole / e Å^{-3}	1.74/-0.94
Flack parameter	0.11(3)

Table S2 Bond length of Eu(TTA)$_3$-L$_2$

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Eu1	O9	2.39(3)	Eu2	O1	2.36(3)
Eu1	O10	2.37(2)	Eu2	O1AA	2.31(3)
Eu1	O13	2.36(2)	Eu2	O8	2.36(3)
Eu1	O14	2.34(2)	Eu2	O15	2.39(2)
Eu1	O16	2.35(2)	Eu2	O46	2.31(3)
Eu1	O17	2.41(2)	Eu2	N19	2.62(3)
Eu1	N24	2.57(3)	Eu2	N35	2.57(3)
Eu1	N31	2.60(3)	S1AA	C6AA	1.511(17)
S6	C8	1.63(3)	S1AA	C9AA	1.457(17)
S6	C41	1.71(4)	S2	C1BA	1.67(4)
S11	C10	1.82(3)	S2	C5AA	1.68(4)
S11	C21	1.80(3)	S4	C3	1.77(4)
F32	C136	1.38(4)	S4	C44	1.73(5)
F37	C136	1.25(4)	F40	C89	1.45(4)
F48	C96	1.34(4)	F43	C89	1.28(4)
F52	C75	1.38(4)	F49	C127	1.41(4)
F54	C136	1.27(4)	F51	C94	1.23(4)
F56	C96	1.41(4)	F58	C89	1.48(4)
F64	C75	1.70(4)	F76	F95	1.71(3)
F69	C75	1.39(4)	F76	C127	1.31(4)
F73	C96	1.42(4)	F84	C94	1.26(4)
O9	C39	1.24(4)	F95	C127	1.02(4)
O10	C66	1.25(4)	F107	C94	1.34(4)
O13	C28	1.26(4)	O0AA	C7AA	1.23(4)
O14	C80	1.35(4)	O1	C8AA	1.22(4)
O16	C57	1.26(4)	O1AA	C30	1.39(5)
O17	C50	1.26(4)	O8	C25	1.28(5)
N14	C88	1.36(4)	O15	C79	1.19(4)
N24	C55	1.31(4)	O46	C4AA	1.32(4)

N24	C71	1.37(4)	N9	C5BA	1.51(4)
N31	C16	1.28(4)	N19	C53	1.43(4)
N31	C34	1.28(4)	N19	C60	1.29(4)
C1A	C6	1.35(4)	N35	C4BA	1.37(4)
C1A	C86	1.37(4)	N35	C36	1.49(5)
C1AA	C15	1.95(4)	C0AA	C6AA	2.01(2)
C1AA	C21	1.74(4)	C0AA	C9AA	1.42(2)
C4	C70	1.58(5)	C0AA	C18	1.455(18)
C4	C81	1.51(5)	C1	C7AA	1.40(5)
C6	C16	1.43(4)	C1	C8AA	1.40(6)
C6BA	C81	1.26(4)	C1BA	C7AA	1.46(5)
C6BA	S0AA	1.41(4)	C1BA	C0BA	1.52(5)
C8	C111	1.62(5)	C2	C5AA	1.32(5)
C10	C15	1.15(4)	C2	C0BA	1.91(4)
C21	C66	1.36(5)	C2AA	C3	1.14(5)
C26	C39	1.45(5)	C2AA	C3AA	1.34(4)
C26	C80	1.30(5)	C2BA	C3BA	1.42(4)
C28	C41	1.46(5)	C2BA	C77	1.43(4)
C28	C62	1.53(5)	C3	C3AA	2.03(5)
C34	C55	1.53(5)	C3AA	C44	1.31(5)
C34	C86	1.38(5)	C3BA	C4BA	1.29(4)
C39	C70	1.48(5)	C4AA	C68	1.28(5)
C41	C98	1.44(5)	C4AA	C94	1.63(6)
C50	C62	1.45(5)	C5BA	C74	1.06(4)
C50	C96	1.45(5)	C5BA	C77	1.71(5)
C55	C93	1.35(5)	C6AA	C18	1.453(18)
C57	C90	1.25(5)	C6AA	C25	1.73(5)
C57	C136	1.68(5)	C8AA	C127	1.63(5)
C66	C90	1.47(5)	C25	C85	1.38(6)
C70	S0AA	1.67(4)	C30	C44	1.46(5)
C71	C122	1.34(5)	C30	C68	1.24(5)
C75	C80	1.42(5)	C36	C53	1.36(5)
C81	S0AA	2.19(3)	C36	C77	1.38(5)
C86	C88	1.33(4)	C53	C87	1.48(5)
C88	C126	1.64(4)	C60	C128	1.46(5)
C93	C125	1.50(4)	C74	C87	1.33(4)
C93	C126	1.44(4)	C78	C87	1.37(4)
C98	C111	1.48(4)	C78	C128	1.32(4)
C122	C125	1.46(4)	C79	C85	1.52(5)
Eu2	O0AA	2.39(3)	C79	C89	1.42(5)

Table S3 Bond angle of Eu(TTA)$_3$-L$_2$

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O9	Eu1	O17	117.5(8)	O1AA	Eu2	O0AA	73.1(9)
O9	Eu1	N24	76.7(9)	O1AA	Eu2	O1	115.8(10)
O9	Eu1	N31	76.0(8)	O1AA	Eu2	O8	76.5(9)
O10	Eu1	O9	136.7(8)	O1AA	Eu2	O15	144.5(9)
O10	Eu1	O17	79.7(8)	O1AA	Eu2	O46	72.3(9)
O10	Eu1	N24	109.9(9)	O1AA	Eu2	N19	139.2(9)
O10	Eu1	N31	70.7(9)	O1AA	Eu2	N35	80.4(10)
O13	Eu1	O9	72.6(9)	O8	Eu2	O0AA	136.2(9)
O13	Eu1	O10	76.3(9)	O8	Eu2	O1	151.3(9)
O13	Eu1	O17	72.5(8)	O8	Eu2	O15	74.1(9)
O13	Eu1	N24	138.0(9)	O8	Eu2	N19	107.4(10)
O13	Eu1	N31	82.6(9)	O8	Eu2	N35	69.7(10)
O14	Eu1	O9	72.3(9)	O15	Eu2	N19	69.9(9)
O14	Eu1	O10	150.1(8)	O15	Eu2	N35	107.1(9)
O14	Eu1	O13	115.4(9)	O46	Eu2	O0AA	119.5(9)
O14	Eu1	O16	86.8(8)	O46	Eu2	O1	80.6(9)
O14	Eu1	O17	78.3(9)	O46	Eu2	O8	78.9(9)
O14	Eu1	N24	80.5(9)	O46	Eu2	O15	82.8(9)
O14	Eu1	N31	135.7(9)	O46	Eu2	N19	148.4(9)
O16	Eu1	O9	146.9(8)	O46	Eu2	N35	142.4(9)
O16	Eu1	O10	70.1(8)	N35	Eu2	N19	64.3(10)
O16	Eu1	O13	140.5(8)	C9AA	S1AA	C6AA	87.4(17)
O16	Eu1	O17	81.4(8)	C1BA	S2	C5AA	99.0(18)
O16	Eu1	N24	74.7(9)	C44	S4	C3	88.5(19)
O16	Eu1	N31	104.6(8)	C127	F76	F95	36.7(17)
O17	Eu1	N24	148.8(8)	C127	F95	F76	50(3)
O17	Eu1	N31	145.0(8)	C7AA	O0AA	Eu2	139(3)
N24	Eu1	N31	62.5(9)	C8AA	O1	Eu2	132(3)
C8	S6	C41	97.5(19)	C30	O1AA	Eu2	133(2)
C21	S11	C10	91.6(14)	C25	O8	Eu2	138(3)
C39	O9	Eu1	136(2)	C79	O15	Eu2	131(2)
C66	O10	Eu1	139(2)	C4AA	O46	Eu2	133(2)
C28	O13	Eu1	138(2)	C53	N19	Eu2	118(2)
C80	O14	Eu1	135(2)	C60	N19	Eu2	125(3)
C57	O16	Eu1	135(2)	C60	N19	C53	118(3)
C50	O17	Eu1	129(2)	C4BA	N35	Eu2	120(2)
C55	N24	Eu1	123(2)	C4BA	N35	C36	120(3)

C55	N24	C71	116(3)	C36	N35	Eu2	120(2)
C71	N24	Eu1	119(2)	C9AA	C0AA	C6AA	71.0(16)
C16	N31	Eu1	124(2)	C9AA	C0AA	C18	117(2)
C16	N31	C34	115(3)	C18	C0AA	C6AA	46.1(9)
C34	N31	Eu1	121(2)	C8AA	C1	C7AA	120(4)
C6	C1A	C86	117(3)	C7AA	C1BA	S2	114(3)
C21	C1AA	C15	88.0(19)	C7AA	C1BA	C0BA	124(3)
C81	C4	C70	93(3)	C0BA	C1BA	S2	120(3)
C1A	C6	C16	117(3)	C5AA	C2	C0BA	116(3)
C81	C6BA	S0AA	110(3)	C3	C2AA	C3AA	109(4)
C111	C8	S6	103(2)	C3BA	C2BA	C77	118(3)
C15	C10	S11	120(3)	S4	C3	C3AA	76.5(16)
C10	C15	C1AA	116(3)	C2AA	C3	S4	110(3)
N31	C16	C6	125(3)	C2AA	C3	C3AA	39(2)
C1AA	C21	S11	114.6(18)	C2AA	C3AA	C3	32(2)
C66	C21	S11	116(3)	C44	C3AA	C2AA	121(4)
C66	C21	C1AA	127(3)	C44	C3AA	C3	91(3)
C80	C26	C39	124(4)	C4BA	C3BA	C2BA	119(3)
O13	C28	C41	119(3)	O46	C4AA	C94	114(3)
O13	C28	C62	126(3)	C68	C4AA	O46	123(4)
C41	C28	C62	115(3)	C68	C4AA	C94	124(4)
N31	C34	C55	118(3)	C3BA	C4BA	N35	125(3)
N31	C34	C86	125(3)	C2	C5AA	S2	110(2)
C86	C34	C55	116(3)	N9	C5BA	C77	114(2)
O9	C39	C26	125(4)	C74	C5BA	N9	115(3)
O9	C39	C70	112(3)	C74	C5BA	C77	130(3)
C26	C39	C70	123(4)	S1AA	C6AA	C0AA	85.5(14)
C28	C41	S6	117(3)	S1AA	C6AA	C25	115(2)
C98	C41	S6	117(3)	C18	C6AA	S1AA	131.7(18)
C98	C41	C28	125(4)	C18	C6AA	C0AA	46.2(9)
O17	C50	C62	133(4)	C18	C6AA	C25	113(2)
O17	C50	C96	111(3)	C25	C6AA	C0AA	159(2)
C96	C50	C62	115(3)	O0AA	C7AA	C1	124(4)
N24	C55	C34	114(3)	O0AA	C7AA	C1BA	120(3)
N24	C55	C93	126(3)	C1	C7AA	C1BA	116(4)
C93	C55	C34	120(3)	O1	C8AA	C1	132(4)
O16	C57	C136	108(3)	O1	C8AA	C127	111(3)
C90	C57	O16	131(4)	C1	C8AA	C127	115(3)
C90	C57	C136	121(3)	C0AA	C9AA	S1AA	115(2)
C50	C62	C28	115(3)	C6AA	C18	C0AA	87.7(15)

O10	C66	C21	119(4)	O8	C25	C6AA	114(3)
O10	C66	C90	122(4)	O8	C25	C85	125(4)
C21	C66	C90	119(4)	C85	C25	C6AA	121(4)
C4	C70	S0AA	108(3)	O1AA	C30	C44	110(3)
C39	C70	C4	126(3)	C68	C30	O1AA	121(3)
C39	C70	S0AA	126(3)	C68	C30	C44	129(4)
C122	C71	N24	128(3)	C53	C36	N35	116(3)
F52	C75	F64	87(2)	C53	C36	C77	131(4)
F52	C75	F69	121(3)	C77	C36	N35	113(3)
F52	C75	C80	113(3)	C3AA	C44	S4	101(3)
F69	C75	F64	114(3)	C3AA	C44	C30	135(4)
F69	C75	C80	117(3)	C30	C44	S4	122(3)
C80	C75	F64	99(3)	N19	C53	C87	123(3)
O14	C80	C75	111(3)	C36	C53	N19	122(4)
C26	C80	O14	126(3)	C36	C53	C87	115(3)
C26	C80	C75	121(4)	N19	C60	C128	120(3)
C4	C81	S0AA	89(2)	C30	C68	C4AA	134(4)
C6BA	C81	C4	125(3)	C5BA	C74	C87	122(4)
C6BA	C81	S0AA	36.9(19)	C2BA	C77	C5BA	136(3)
C1A	C86	C34	119(3)	C36	C77	C2BA	124(3)
C88	C86	C1A	108(3)	C36	C77	C5BA	100(3)
C88	C86	C34	132(3)	C128	C78	C87	122(3)
N14	C88	C126	123(2)	O15	C79	C85	133(4)
C86	C88	N14	127(3)	O15	C79	C89	116(3)
C86	C88	C126	109(2)	C89	C79	C85	110(3)
C57	C90	C66	122(4)	C25	C85	C79	119(4)
C55	C93	C125	117(3)	C74	C87	C53	120(3)
C55	C93	C126	122(3)	C74	C87	C78	126(3)
C126	C93	C125	121(3)	C78	C87	C53	114(3)
F48	C96	F56	107(3)	F40	C89	F58	90(2)
F48	C96	F73	98(3)	F43	C89	F40	108(3)
F48	C96	C50	118(4)	F43	C89	F58	112(3)
F56	C96	F73	115(3)	F43	C89	C79	114(3)
F56	C96	C50	109(3)	C79	C89	F40	111(3)
F73	C96	C50	111(3)	C79	C89	F58	119(3)
C41	C98	C111	102(3)	F51	C94	F84	125(4)
C98	C111	C8	116(2)	F51	C94	F107	103(3)
C71	C122	C125	115(3)	F51	C94	C4AA	108(4)
C122	C125	C93	117(3)	F84	C94	F107	97(3)
C93	C126	C88	120(3)	F84	C94	C4AA	112(3)

F32	C136	C57	109(3)	F107	C94	C4AA	109(3)
F37	C136	F32	105(3)	F49	C127	C8AA	106(3)
F37	C136	F54	130(3)	F76	C127	F49	91(3)
F37	C136	C57	107(3)	F76	C127	C8AA	105(3)
F54	C136	F32	97(3)	F95	C127	F49	128(3)
F54	C136	C57	106(3)	F95	C127	F76	93(3)
O0AA	Eu2	O15	142.3(8)	F95	C127	C8AA	123(4)
O0AA	Eu2	N19	78.0(9)	C78	C128	C60	122(3)
O0AA	Eu2	N35	74.6(9)	C6BA	S0AA	C70	102(2)
O1	Eu2	O0AA	72.0(9)	C6BA	S0AA	C81	32.6(16)
O1	Eu2	O15	83.6(9)	C70	S0AA	C81	69.5(16)
O1	Eu2	N19	80.7(10)	C1BA	C0BA	C2	89(2)
O1	Eu2	N35	135.6(10)				