Supporting Information

METHODS

Materials and Instrumentation. All reaction materials were obtained from commercial suppliers, and used without further purification. UV-Vis absorption spectra were recorded using a Shimadzu UV-2450 spectrophotometer. Fluorescence spectra and decay lifeime were measured on an Edinburgh FLS980 or FLS5 Photoluminescence Spectrometer. PXRD measurements were performed on an X-ray powder diffractometer (Rigaku, Japan) at 4 kW, 3 mA.

Synthesis of Ligand. The ligand H_2hpi2cf was prepared according to our previous report.[S1]

Synthesis of LIFM-42(Nd) and LIFM-42(Gd). $Ln(NO_3)_2 \cdot 6H_2O$ (0.1 mmol, Ln = Nd or Gd) H_2hpi2cf (0.1 mmol, 0.053 g), and 4 mL DMF/H_2O (v/v = 1:1) were added into a 10 mL Teflon cup. The mixture was incased into the matched stainless steel autoclave. The sealed autoclave was heated at 100 ^oC in the oven for 50 hours and cooled to the room temperature at the rate of 10 ^oC h^{-1}. Block crystals of **LIFM-42(Nd) and LIFM-42(Gd)** were gained by filtration and dried in vacuum.

Single-crystal X-Ray crystallography. Single-crystal diffraction data of **LIFM-42**(Nd) were collected on a Rigaku Oxford Gemini S Ultra diffractometer with the Enhance X-ray Source of Cu-Kα radiation (λ = 1.54178 Å) at 150 K. Absorption corrections were applied using multiscan technique. The crystal structures were solved by direct method of SHELXS and refined against F^2 using the SHELXL programs. The non-hydrogen atoms were treated with anisotropic parameters, and H atoms were placed in calculated positions and refined using a riding model. Crystallographic data and structural refinement information were listed in Table S1, and deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 1821282.

Reference

[S1] L. Chen, S.-Y. Yin, M. Pan, K. Wu, H.-P. Wang, Y.-N. Fan and C.-Y. Su, *J. Mater. Chem. C*, 2016, **4**, 6962-6966.

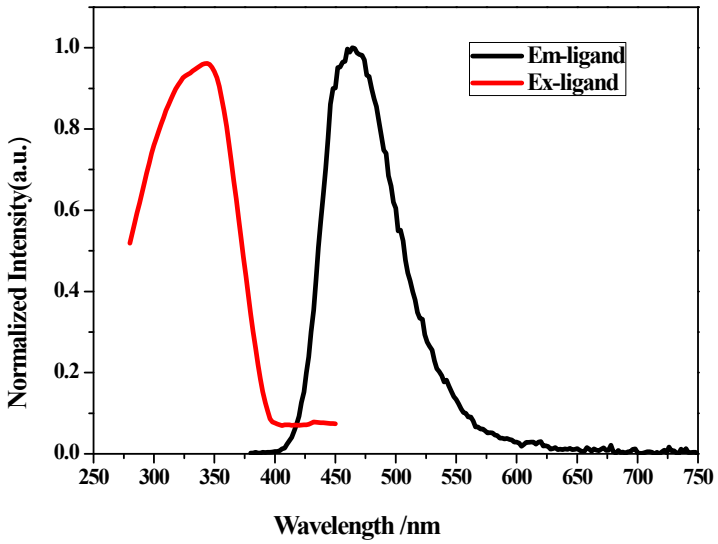

Fig. S1 Excitation (red) and emission (black) spectra of the ligand in DMF solution (1×10^{-5} mol L^{-1}, RT).

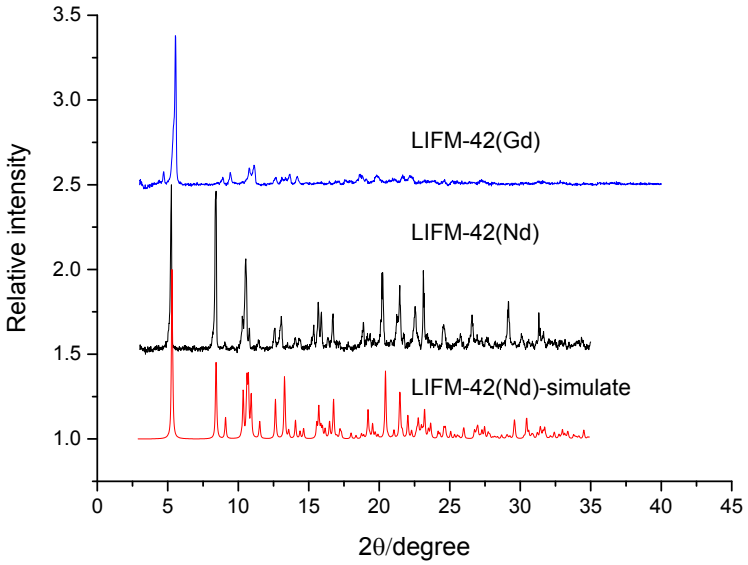

Fig. S2 PXRD patterns of LIFM-42(Nd) and LIFM-42(Gd) compared with simulated.

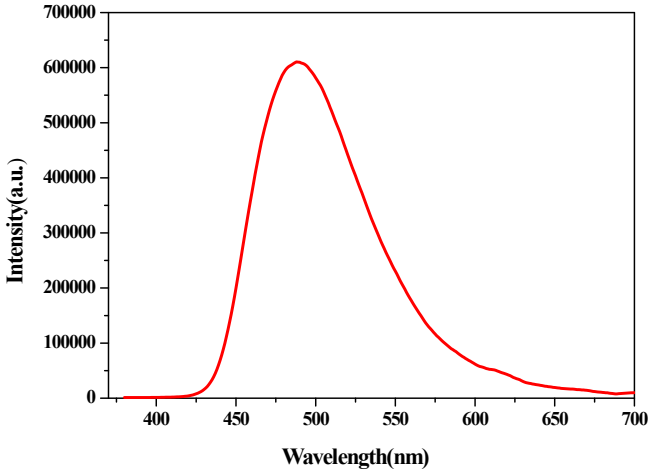

Fig. S3 Emission spectra of LIFM-42(Gd) at 77 K (λ_{ex} = 340 nm).

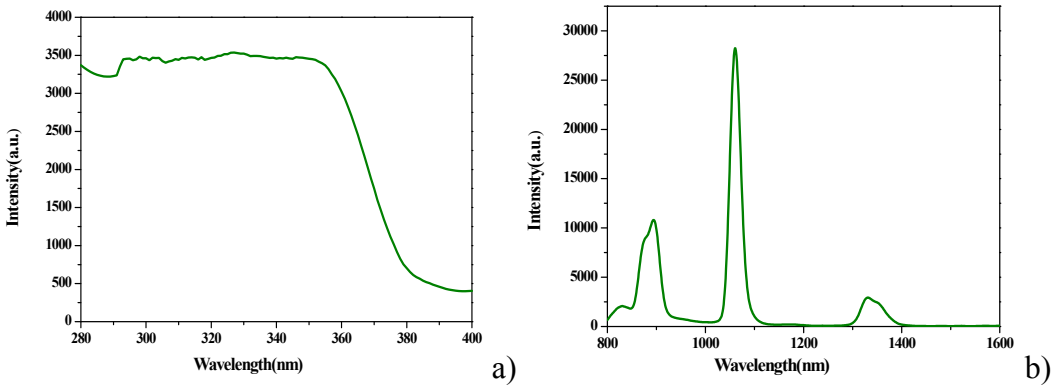

Fig. S4 Solid state excitation (a, λ_{em} = 1060 nm) and emission (b, λ_{ex} = 360 nm) spectra of **LIFM-42**(Nd) at RT.

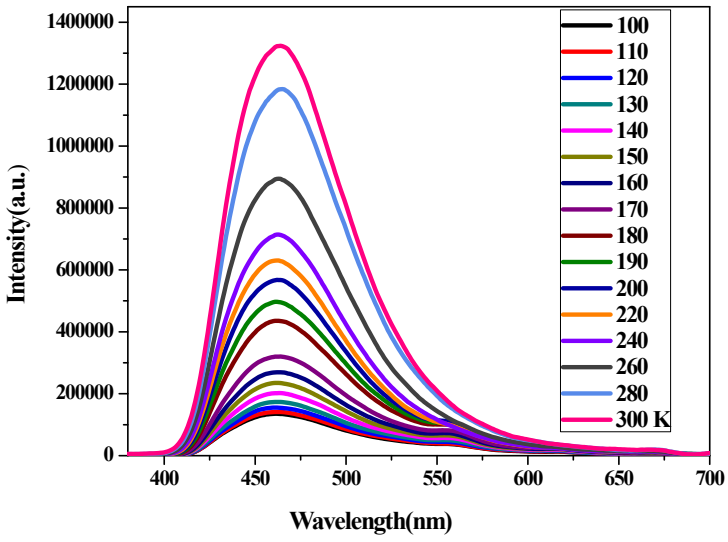

Fig. S5 Variable temperature (VT) phosphorescence of Gd-complex by dipping a drop of acetone.

Table S1 Crystallographic data for **LIFM-42**(Nd).

Compound	**LIFM-42(Nd)**
CCDC No.	1821282
Formula	$NdC_{36}H_{32}N_4O_{10}F_3$
Formula Weight	881.89
Shape / Color	Block/Colorless
Crystal System	Triclinic
Space Group	*P*-1
T (K)	150
a (Å)	10.1306(5)
b (Å)	11.4102(6)
c (Å)	17.3413(7)
α / β / γ (°)	70.926(4) /88.066(4) /72.035(4)
V (Å^3)	1797.08(16)
Z	2
D_{calc} (g/cm^3)	1.630
μ (mm^{-1})	11.717
F (000)	886
R_{int}	0.0470
Reflections collected / unique	10543/ 6225
Completeness	99.6%
Data / Restraints / parameters	6225/0/493
R_1 [I>2σ(I)]	0.0394
wR_2 (all data)	0.0985
GOF	1.034